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SOME CHARACTERISTICS OF FRESH PORTLAND CEMENT PASTES AND MORTARS

by

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The undersigned certify that they have read, and recommend to the Faculty of Graduate Studies for acceptance, a thesis entitled SOME CHARACTERISTICS OF FRESH PORTLAND CEMENT PASTES AND MORTARS submitted by GEORGE (JIRI) STASTNY in partial fulfilment of the requirements for the degree of Master of Science.

ABSTRACT

The purpose of this investigation was to use continuous testing methods to study certain properties of Portland cement pastes and mortars from the start of mixing through the setting phase and into the hardened state. Two methods were used; the continuous workability test using the Halliburton Thickening Time Tester and an electrical test based on the electrical cell principle where cement paste performed as the electrolyte.

The investigation showed that both test methods are able to follow certain characteristics of cement pastes and mortars during this period. The continuous workability test showed interesting characteristics concerning various types and sources of cement and the electrical test indicated its possibility as an alternative test to the Vicat procedure for determining the initial set time of cement pastes.

The attainment of study objectives in this program was limited by the lack of available methods with which to compare the observations as a means of interpreting the results. Consequently this study results largely in recommendations for future research and studies to investigate the term "workability" and test methods to relate laboratory investigations to the "field concept of workability." Further studies are recommended to expand the possibilities of the electrical test to explain its nature and origin relative to chemical and mineralogical changes which take place during the setting and hardening process.

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CHAPTER I

INTRODUCTION

Studies on setting properties of cement were initiated decades ago and a number of methods are available to test various characteristics of cement paste, mortar and concrete during the setting stage. Most of these methods give results indicating conditions at the time of test and reveal little in the way of continuous information about the nature or extent of the setting properties of the above materials. Only a few methods enable one to follow the setting of cement continuously, such as the classical work of Lerch (1946) which follows properties and changes of cement paste through heat liberation pattern studies or the continuous X-ray diffraction studies done by Seligman and Greening (1964). These continuous observation procedures involve sophisticated equipment and techniques not readily available to the ordinary laboratory.

It was the purpose of this program to study and develop methods that might be employed using simpler procedures and techniques that would yield significant information during the setting interval of Portland Cements. On the basis of literature studies it seemed that a study of some electrical characteristic of the setting cement might be suitable.

Several studies have been made to develop methods utilizing electrical resistivity measurements of cement paste, mortar or concrete (Monfore, 1968; Freitag, 1959).

The only work* known to the author utilizing the idea of using cement paste as the electrolyte to create an electrical cell was done in Finland in the late 1950's.

An investigation by the author on the use of the electrical test started about four years ago in Czechoslovakia when the concept of the electrical cell was used to get additional information on aerated light-weight concrete. None of this work has been published to date nor are the results now available to the author. The work reported here is a continuation of the above using Portland cement paste as the electrolyte and using lead and copper as electrodes.

A limitation of this work was the Vicat time of set test used for comparison with the electrical test results. The Vicat test is limited to giving a mechanical condition of the cement paste at a given instant in time. It was considered that some type of "workability test" could be another useful basis of comparison of results especially during the interval of time immediately following mixing and prior to time of set by the Vicat method. Since consistency is resistance of a substance to deformation and is one of the properties that influences workability, it was considered that the Halliburton Consistometer, developed for testing of oil well cements, might be useful for this purpose.

Consistency of cement pastes with the Halliburton Consistometer is reported as viscosity in poises because this unit was considered preferable to the equipment scale reading in terms of an "index of

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*The work referred to came to the author's attention while employed as a researcher with the Research Institute of Building Materials in Brno, Czechoslovakia and, unfortunately, the reference is no longer available to him, nor can it be identified.

torque." The conversion from "index of torque" to viscosity in poises was made in accordance with the procedure recommended by the equipment developer. A serious limitation of this method was the restricted cement paste water content which was necessary to use to meet the apparatus range of consistency measurement.

CHAPTER II

FUNDAMENTAL THEORY RELATED TO PORTLAND CEMENT PASTE PROPERTIES AT EARLY STAGES

2.1 Definition of Portland Cement

Portland cement is a finely powdered material, usually gray or brownish gray, consisting of artificial crystalline minerals, the most important of which are the calcium silicates. These react with water and produce new mineral compounds that are able to set and harden and to develop considerable strength. The reaction product is of low permeability to water, and is nearly insoluble in water.

2.2 Chemical and Mineralogical Composition of Portland Cement

The two most important mineral compounds are two calcium silicates: tricalcium silicate (C_3S) called alite, and β-dicalcium silicate (β-C_2S) called belite. These two silicates, which constitute about 75 per cent of Portland cement by weight, are embedded in a mixed crystalline interstitial material composed of solid solution of lime, alumina, and ferric oxide; this composition has been generally known as tetracalciumaluminoferrite (C_4AF) and as an important mineral tricalciumaluminate (C_3A). This iron-containing phase in Portland cement is simply referred to as the "ferrite phase" (Taylor, Copeland, and Kantro, 1964).

It can be seen from the above mineralogical composition that four compounds are usually regarded as the major chemical constituents of Portland cement: calcium oxide (CaO), silica oxide (SiO_2), aluminum oxide (Al_2O_3), and ferric oxide (Fe_2O_3).

In reality, the silicates in cement are not pure compounds of the above noted oxides but contain minor oxides in solid solution. These minor oxides have significant effects on the atomic arrangements, crystal forms, and hydraulic properties of the silicates. The minor compounds, such as MgO, K_2O and Na_2O, TiO_2, and Mn_2O_3 usually amount to not more than a few per cent of the weight of Portland cement.

The oxides of sodium and potassium are of the greatest interest. They have been known to react with some aggregates in concrete, they have been found to affect the rate of strength of cement (Neville, 1959), they have been observed to influence the setting time of cement paste if present in large amounts (Czernin, 1962), and they seem to promote "flash set" of cement paste (Taylor, Copeland, and Kantro, 1964).

In addition, there are two other types of constituents in Portland cement generally included in the categories of "loss on ignition" and "insoluble residue." They represent substances that can be expelled from cement by heating (water and carbon dioxide) in the first and the fraction of cement that is insoluble in hydrochloric acid (clayey compounds, usually) in the second case. A higher "ignition loss" may have adverse effects on the hardening properties of cement paste and the amount of "insoluble residue" can indicate the completeness of clinkering reactions during production of Portland cement.

Sulphuric anhydride is an important minor constituent of Portland cement derived from calcium sulphate-gypsum ($CaSO_4.2H_2O$). Gypsum is added to Portland cement to regulate its setting time. The amount of gypsum has to be controlled carefully. Too small an amount can result in a "flash set" of the alumina compound in Portland cement (C_3A), too large an amount may cause an expansion of cement paste due to sulphates. The action of gypsum and the alumina compound hydration control will be discussed later.

2.3 Hydration and Chemical Behavior of Products of Hydration of Portland Cement.

Reactions of the Portland cement compounds and water are basically of two types: true hydration and hydrolysis. It is usual, however, to apply the term hydration to all reactions that occur when cement is mixed with water (Neville, 1965).

The initial reactions of Portland cement with water are rather of a violent character. The reaction of the tricalciumaluminate (C_3A) phase alone with water is immediate and a formation of crystalline hydrate occurs rapidly with liberation of a large amount of heat. It can cause a "flash set" of Portland cement, unless the rapid reaction is moderated by some means, i.e. by addition of gypsum that will react with tricalciumaluminate (C_3A) to form the relatively insoluble sulphoaluminates that act as a coating on the tricalciumaluminate (C_3A) phase to slow down the reaction.

There are two basic types of calcium aluminosulphate hydrates formed in the hydrating process. The "high sulphate" form, generally

called "ettringite," can be represented as $3CaO.Al_2O_3.3CaSO_4.30\text{-}32H_2O$ and the "low sulphate" form can be represented as $3CaO.Al_2O_3.CaSO_4.12H_2O$. Even today the details of formation of the sulphoaluminates are not quite clear because of the complexity of the reactions during the early hydration period of cements and because of different compositions of cements (Troxell, Davis and Kelly, 1968).

Gypsum reacts not only with tricalcium-aluminate (C_3A); with tetracalcium aluminoferrite (C_4AF) it forms calcium sulphoferrite and its presence may accelerate the hydration of silicates. The optimum content of gypsum can be determined by observation of the heat of hydration and by the soundness test of Portland cement.

The tricalcium silicate (C_3S) and β-dicalcium silicate ($\beta\text{-}C_2S$) are the two most important constituents of Portland cement. There are several interesting aspects that should be noted. Both tricalcium silicate and $\beta\text{-}C_2S$, respectively result in the formation of tricalcium disilicate hydrate ($C_3S_2H_3$) as the main final product of reactions. The tricalcium silicate is transformed as follows:

$$2(C_3S) + 6H \longrightarrow C_3S_2H_3 + 3Ca(OH)_2 \qquad [1]$$

and this reaction is rapid and takes place during the early stages of cement hydration. The dicalcium silicate reaction can be expressed:

$$2(C_2S) + 4H \longrightarrow C_3S_2H_3 + Ca(OH)_2 \qquad [2]$$

It can be seen clearly from the above calculations that there is not much difference in the amount of water combined by the C_2S [2] and C_3S [1] but the reaction of tricalcium silicate [1] releases much more

free calciumhydrate than is released in the reaction of dicalcium silicate [2].

It has to be considered, however, that the above calculations [1] and [2] assume pure compounds of Portland cement. Recent studies have shown (Taylor, 1964) that the ratio of CaO, SiO_2 and water varies within certain limits. All cements produced commercially have a certain amount of impurities in the calcium silicates, generally Al and/or Mg atoms locked into their molecular structures that are usually carried into hydration reactions (Copeland, Kantro and Verbeck, 1963).

The rates of hydration of pure compounds of Portland cement are shown in FIGURE II.1.

2.4. Physical Properties of Hydration Products of Portland Cement.

Physical properties of the calcium silicate hydrates are important in connection with the setting and hardening of cement paste.

Studies using Calcium 45 tracer have indicated that calcium silicates do not enter the hydration reaction in the solid state but they probably first pass into a solution and then react to form somewhat less soluble hydrate silicates (Spinks, Baldwin and Thorvaldson, 1952). The cement gel formed consists of extremely small crystals in the first hours after mixing of cement with water. As cement hydration continues the gel formation continues to fill the available space or until all cement is hydrated and provides a strong bond between the original cement grains. The specific surface of cement gel has been found to be about 2×10^6 cm^2/gm. (Powers, 1948). Cement gel is a "rigid" gel of

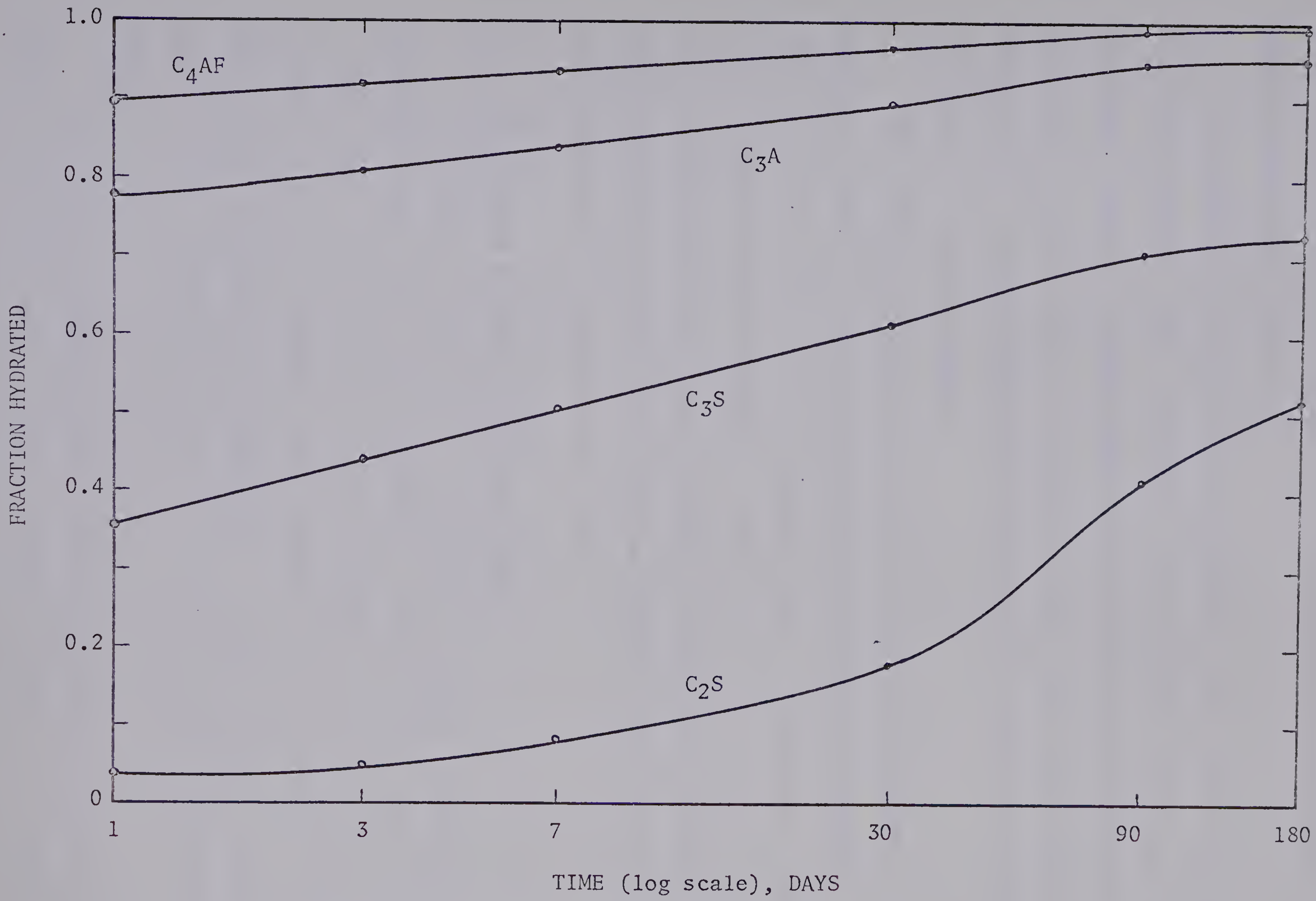

FIGURE II.1 RATE OF HYDRATION OF PURE COMPOUNDS
(after Neville, 19)

high strength and is a colloidal and mainly homogeneous mass that contains chemically bound water.

The quantity of bound water approximates 25 per cent of the weight of cement (Czernin, 1962). Evaporable water occupies about 25 per cent of the gel volume - corresponding to about 15 per cent of the weight of the cement (Czernin, 1962). Although this "gel water" can be freed by drying at 105°C leaving empty pore space it is not capable of hydrating remaining fresh cement grains.

The hydrated calciumsilicates have a molecular arrangement similar to that of a natural mineral called tobermorite, (Brunauer, 1962) and this is often called tobermorite gel. The gel plays a vital role in determining the rheological properties of fresh Portland cement paste, which properties in turn determine the consistency and workability of fresh concrete, and it plays an important role in the setting and hardening of the cement paste as well.

2.5 The Setting Process of Portland Cement.

Setting is the term used to describe the gradual stiffening of the cement paste and it can be defined as a change from a semi-fluid to a rigid state. For practical use it is important that cement pastes remain in a plastic condition for a period of time after mixing or in other words, to preserve its workability. In spite of this "workable" period, quite a rapid reaction sets in as soon as cement is mixed with water.

The first two compounds that enter the reaction are C_3A and C_3S. The C_3A can result, unless properly retarded by gypsum, in an almost

immediate set of cement taking place which is referred as "flash set." If the C_3A component is "normally" retarded the C_3S compounds will largely determine the setting characteristics.

During this period the C_3S is gradually hydrated to "tobermorite" in the form of a gel releasing calcium hydroxide which slowly crystalizes from the solution. The aluminates pass into the solution and react with the dissolved gypsum and precipitate as insoluble calcium sulfoaluminate (ettringite). The dicalcium silicate reacts more slowly.

All the above processes produce gel, and gradual stiffening of the cement paste occurs. Continuing stiffening can be tested and it is possible to follow its development long before the setting time can be determined by the Vicat apparatus.

The stiffening in early stages can be completely overcome by mechanical working. This is a "thixotropic" effect that can be found in many colloidal systems.

FIGURE II.2 shows four basic stages in setting and hardening of Portland cement. It is a simplified diagramatic representation of the possible sequence of cement paste changes. The four stages are:

(a) Dispersion of unreacted Portland cement clinker in water.

(b) After a few minutes: hydration products "eat" into and grow out from the surface of each grain.

(c) After a few hours: the coating of different grains have begun to join up, the gel thus becoming continuous, (setting).

(d) After a few days: further development of the gel has occurred (hardening).

In addition to the phenomena of "flash set" previously referred

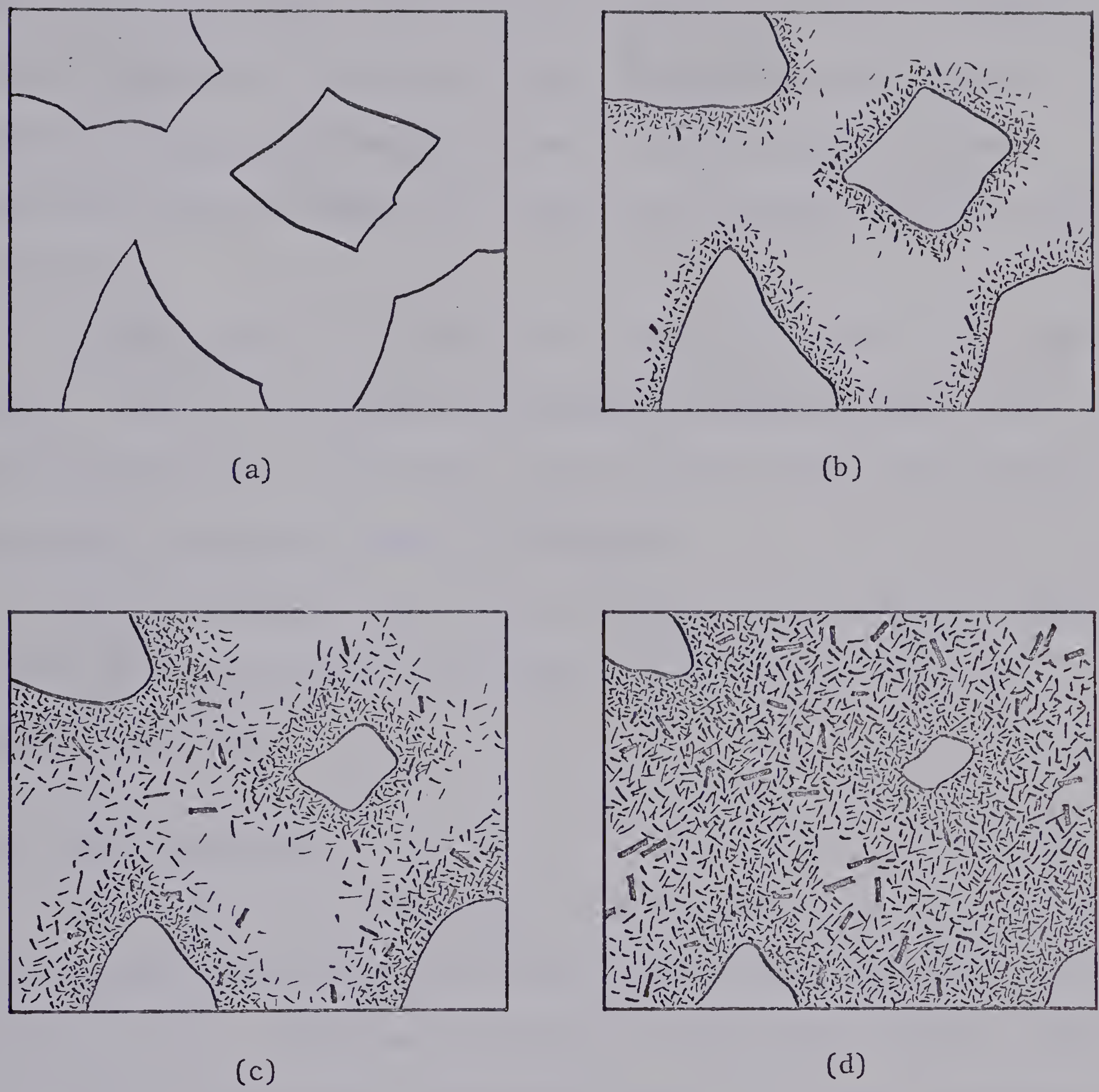

FIGURE II.2 FOUR STAGES IN THE SETTING AND HARDENING OF PORTLAND CEMENT (after Taylor, 1964)

to, occasionally, cements show another type of abnormal premature stiffening several minutes after the addition of mixing water which has been called "false set." This may be caused by the action of gypsum that was dehydrated to plaster of Paris ($CaSO_4.1/2H_2O$) or soluble anhydride ($CaSO_4$) under the conditions of excessively high clinker temperature when the gypsum and clinker were interground during cement manufacture.

Another cause of the "false set" can be the alkali content of cement. Alkalis can carbonate during long storage and react with calcium hydroxide in cement paste. The resulting $CaCO_3$ precipitates and causes a premature rigidity of the paste.

In all cases the "false set" can be overcome by remixing only; to break the comparatively weak skeletons of plaster of Paris or precipitated $CaCO_3$.

2.6 Heat Evolution.

One of the various techniques that have been used to study the early hydration reaction in the cement paste is the heat evolution or liberation study. The first work done by Lerch (1946) resulted in the heat liberation plot that is shown in FIGURE II.3. Three significant heat liberation peaks are clearly visible.

In the first stage that lasts only a few minutes after the first contact of cement with water, there is a brief period of very rapid and high heat evolution. This peak is a result of wetting of cement, solution of gypsum and alkalis, and formation of a sulfoaluminate coating of the C_3A phase to retard a violent hydration reaction that

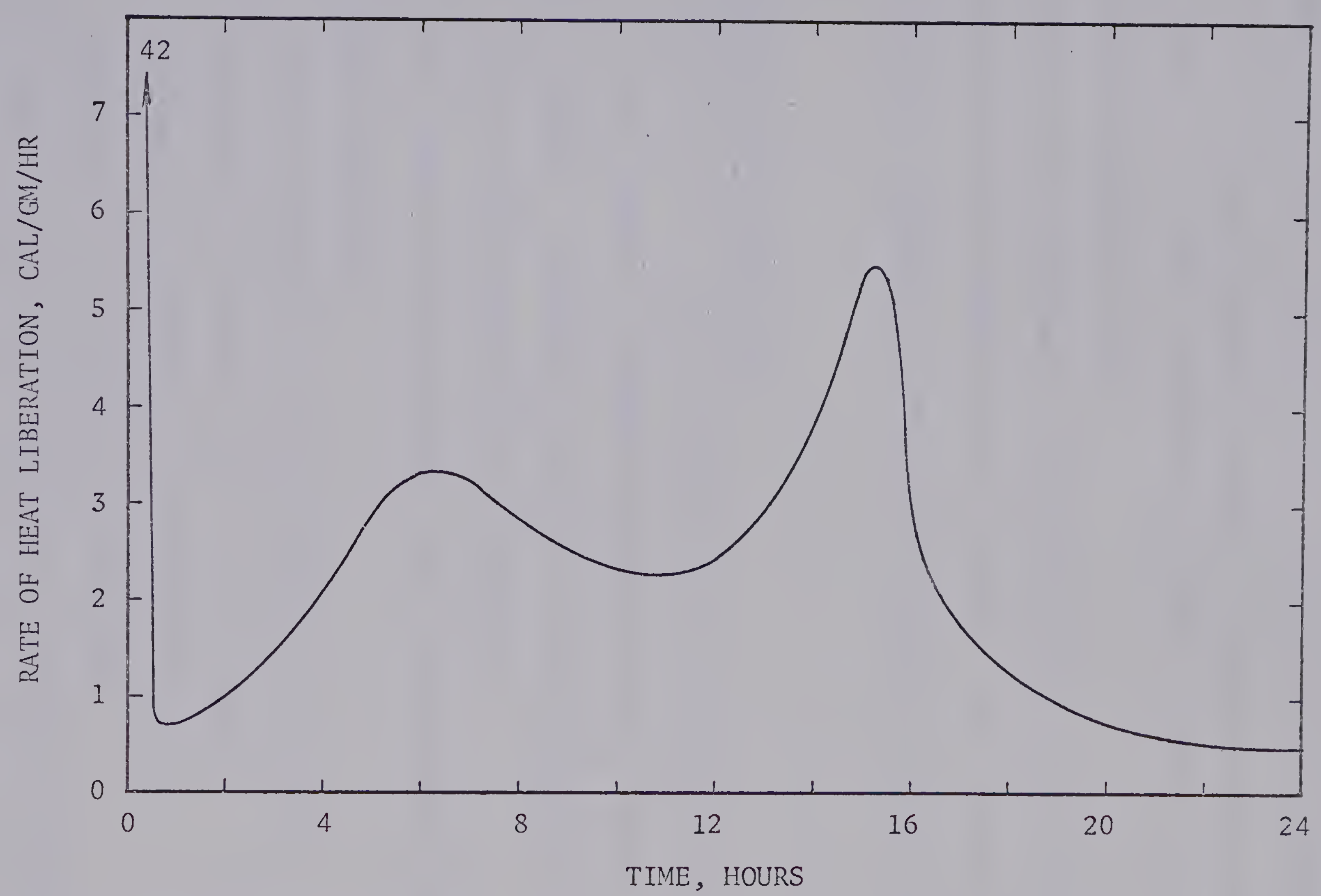

FIGURE II.3 HEAT LIBERATION PATTERN OF A PORTLAND CEMENT (after Verbeck, 1965)

could cause the "flash set" of cement paste. The second heat liberation peak indicates the continued slow hydration of C_3A and the increasingly rapid hydration of C_3S. The third heat evolution peak is commonly observed and its time and magnitude depend on the C_3A, gypsum, and alkali contents. The third peak thus represents a retarded but still rather violent hydration of C_3A.

Recent work by Seligmann and Greening (1964) involved continuous X-ray diffraction of wet cement pastes during the early stages of hydration and has confirmed the reactions assumed by the heat liberation studies.

2.7 Workability of Cement Paste.

Workability is a relative property of fresh cement paste, mortar or concrete that depends on a number of properties which cannot be satisfactorily measured. Workability itself is a broad term which is not, in fact, fixed by general definition or agreement.

Troxell, Davis and Kelly (1968) listed four essential properties that influence workability:

(a) the shear resistance or force required to start flow,

(b) the mobility of the mass after flow has started,

(c) cohesiveness or resistance to segregation, and

(d) stickiness that is related to cohesiveness.

These four properties are further affected by the type and characteristics of cement, water content, chemical admixtures, and by the amount of entrained air.

Glanville, Collins and Matthews (1947) defined workability as the

amount of useful internal work necessary to produce full compaction of the cement paste or concrete.

According to Neville (1965) the main factors affecting workability are water content of the mix, grading of aggregates and the amount of entrained air.

Ney (1963) promotes the resilience (work done in deformation) as the most important element of workability that can be measured by an instrument.

Ohnemuller (1967) developed a new test method to measure workability. He proposed a unit of workability that he defined in terms of deformation work and discussed the results by comparison with the cone penetration test described by Steinegger (1967). Steinegger listed individual properties constituting the concept of workability as follows: cohesion, adhesion, flow, plasticity, dilatancy, thixotropy, desorption of water, water retentivity, setting rate, and pumpability.

It is obvious from the above that the concept of workability of both cement and concrete is rather complex and that presently it cannot be measured by one or even by several test methods now in use.

Cement paste changes its properties with time, and workability is also dependent on the progress of the hydration reaction and on physical aspects of the setting and hardening process.

While cement paste or concrete can be manipulated (placed, transported, remixed, compacted) for a certain period of time after mixing, it stiffens gradually as the hydration process proceeds. The gel formation finally reaches the stage, when the particles of the gel make sufficiently close contact and develop an interstructure resulting in initial set.

CHAPTER III

TEST PROGRAM AND PROCEDURES

3.1 General.

The object of this program was to take a new look at the properties and behavior of cement pastes at early stages after mixing. To accomplish this objective two basic types of test were run, namely workability and electrical test.

3.2 The Workability Test.

3.2.1 General Description

The Halliburton Thickening Time Tester, model 800.5 was used for the workability test of cement pastes. The apparatus is based on consistency measurement in which resistance of cement paste is measured in units of torque thus providing a laboratory means for a study of setting characteristics and viscosity of cement pastes.

3.2.2 Materials

Four types of cement all obtained from local producers in Edmonton were tested:

Inland Normal Portland Cement,

Inland High Early Strength Cement,

Inland Sulphate Resistant Cement, and

Canada Normal Portland Cement.

3.2.3 Preparation of Specimens.

Cement and water were mixed according to ASTM C 305-65, Standard Specification for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency, in the three speed-paddle-type mixer. The water content was selected so that the viscosities of tested cement pastes were within the range limitations of the testing equipment; 20 to 94 poises.

Temperatures of used materials were: 70±2°F for the cement and 70±0.5°F for mixing water. Laboratory conditions were maintained on 70±2°F and 50±1 per cent of relative humidity of air.

The following water/cement ratios were tested:

Inland Normal Portland Cement - 0.34, 0.35, 0.40 and 0.42,

Inland High Early Strength Cement - 0.40, 0.45, 0.47 and 0.50,

Inland Sulphate Resistant Cement - 0.33, 0.34, 0.35 and 0.37,

Canada Normal Portland Cement - 0.32, 0.33, 0.34 and 0.35.

3.2.4 Laboratory Equipment.

The apparatus used for workability testing of cement pastes was the Halliburton Thickening Time Tester, model 800.5, which was developed for testing oil well cements. The apparatus gave cement consistency in units of "index of torque." By means of a calibration test these units can be converted to poises. The consistency of the cement paste in poises was plotted with respect to time. Detailed description of the apparatus is given in Appendix A.

3.2.5 Testing Procedure.

Detail description of the testing and calibration procedures is

given in Appendix A.

3.3 Electrical Test on Cement Paste and Mortar.

3.3.1 General Description

This test is based on the principle of an ordinary electrical cell. Two different metals, lead and copper, were used as electrodes and fresh cement paste or mortar as the electrolyte. An electrical output, voltage, from this "cell" was recorded by a voltmeter, while simultaneously run specimens were tested by standard methods to determine the initial setting time of the cement paste or mortar. The recorded electrical output was compared with the standard method results.

3.3.2 Materials

Two types of Portland cement, both from Edmonton producers were tested:

Inland Normal Portland Cement, and

Canada Normal Portland Cement.

Standard sand 20-30 was used as aggregate and Canada Normal Portland Cement as binder for the electrical testing of setting properties of cement mortars.

3.3.3 Preparation of Specimens

Testing was done on Portland cement pastes of normal consistency as determined by ASTM test procedure C 187-64; Standard Method of Test of Normal Consistency of Hydraulic Cement. The paste for the electrical testing of setting characteristics was prepared by hand mixing according to former ASTM 187-52; Standard Method of Test of Normal Consistency of

Hydraulic Cement.

Cement mortars were also hand mixed according to ASTM procedures C 190-63; Standard Method of Test for Tensile Strength of Hydraulic Cement Mortars.

Mortars were prepared and the water content determined according to ASTM C 190-63; Standard Method of Test for Tensile Strength of Hydraulic Cement Mortars.

3.3.4 Laboratory Equipment

The Vicat apparatus (ASTM C 191-65; Time of Setting of Hydraulic Cement by Vicat Needle) was used for the test of setting time of the cement paste.

Electrodes for the electrical test were made in the Civil Engineering Shop at the University of Alberta. They consisted of two metal electrodes, lead and copper, separated by plexiglas. The "AO" type of electrode is shown in FIGURE III.1.

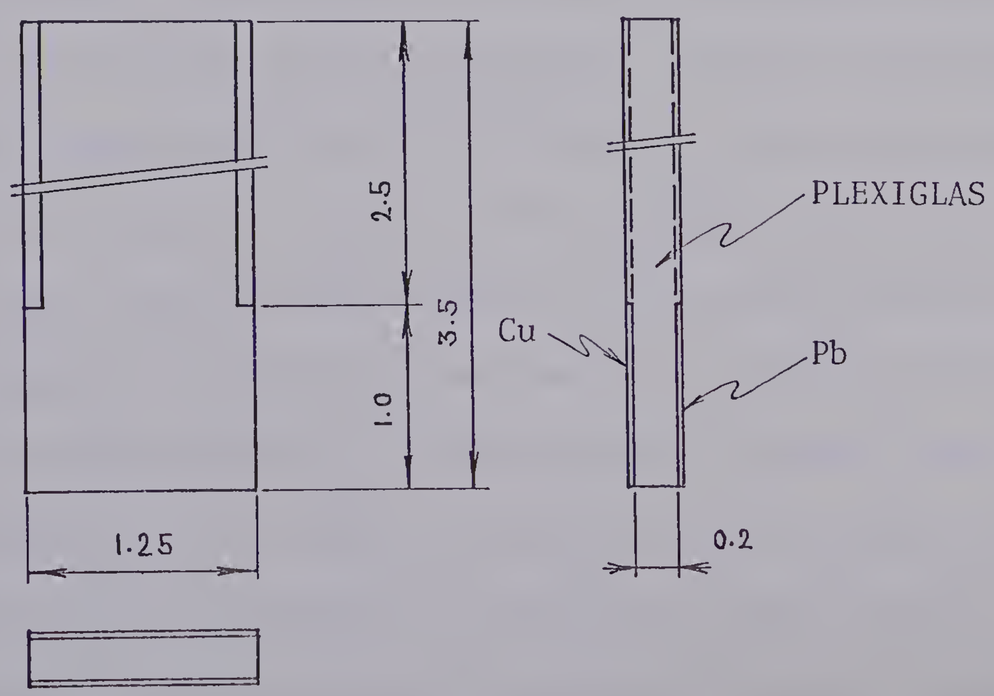

FIGURE III.1 "AO" TYPE ELECTRODE

The electrical output from the electrical cell was recorded by recording voltmeter Moseley Autograf, model 680. The speed of the recording tape was 1 inch per hour, range of voltage chosen for the test was 0 to 1 Volt.

Five types of electrodes were tested to select the best performing electrode. A description of the five types of electrodes used is in Appendix B.

The soil penetrometer described in ASTM D 1558-63 (Standard Method of Test for Moisture-Penetration Resistance Relations of Fine-Grained Soils) was used to determine the initial setting time of cement mortars tested. The procedure to establish the setting time of the mortars was in accordance with ASTM C 403-63T (Tentative Method of Test for Time of Setting of Concrete Mixtures by Penetration Resistance).

3.3.5 Testing Procedure

The cement paste of normal consistency was placed in a Vicat ring after proper mixing. Electrodes were rinsed in hydrochloric acid (one part of HCl and two parts of distilled water), washed in distilled water and dried then immediately placed in the prepared cement paste and connected to the recording voltmeter. The specimens were tested in the Vicat rings under controlled conditions of 70°F and 50% relative humidity. One specimen of cement paste was tested for setting time by Vicat apparatus, the second specimen was tested by the electrical test.

Cement mortars were tested in a similar manner except that the initial setting time was determined according to ASTM C 403-63T (Tentative Method of Test for Time of Setting of Concrete Mixtures by Penetration Resistance.)

CHAPTER IV

RESULTS

4.1 Workability test

4.1.1 Inland Cements

4.1.1.1 Inland Normal Portland Cement

TABLES IV-1 and IV-2 show the variation of consistency with time for Portland cement pastes of various water/cement ratio prepared from Inland Normal Portland Cement.

TABLE IV-1

CONSISTENCY IN UNITS OF "INDEX OF TORQUE" FOR WATER/CEMENT RATIOS 0.34 AND 0.35

Time (min)	Scale reading (index of torque) for water/cement ratio							
	0.34		0.35					
	Trial number							
	1	2	1	2	3	4	5	6
5	7.2	6.2	5.7	6.0	5.1	5.2	5.1	5.0
15	6.3	5.8			4.7	4.8	4.6	4.6
30	6.4	5.8			4.6	4.7	4.5	4.5
45	6.1	6.1	4.9	5.1	4.7	4.8		
60	6.2	6.7	4.9	5.2	4.8	5.0	4.7	4.9

TABLE IV-1 -- Continued

Time (min)	Scale reading (index of torque) for water/cement ratio							
	0.34		0.35					
	Trial number							
	1	2	1	2	3	4	5	6
75	6.5	6.7	5.1	5.7				
90	7.1	7.2	5.2	6.0	5.3		5.2	5.4
105	7.6	7.9	5.7	6.5	6.1	5.9	5.5	5.9
120	8.3	8.5	6.0	7.0	6.4	6.3	6.0	6.6
135	9.2	9.2			7.1	7.1	6.5	7.2
150	10.0	10.0	7.7	8.7	8.8	8.4	7.8	8.8
165	-	-	9.3	10.0	9.6	9.5	9.0	9.2
180			10.0		10.0	10.0	10.0	10.0
*)	145	145	168	165	170	170	172	172

*The time when scale reading reached the value of 10.0

TABLE IV-2

CONSISTENCY IN UNITS OF "INDEX OF TORQUE" FOR

WATER/CEMENT RATIOS 0.40 AND 0.42

Time (min)	Scale reading (index of torque) for water/cement ratio									
	0.40								0.42	
	Trial number									
	1	2	3	4	5	6	7	8	1	2
5	3.9	3.8	3.7	3.8	3.8	3.8	3.0	3.2	2.4	2.3
15			3.3	3.5	3.3	3.5	3.0	3.0	2.3	2.2
30	3.3	3.2	3.1	3.4	3.2	3.4	2.9	2.9	2.2	2.1
45	3.3	3.2	3.1	3.4					2.2	2.1
60	3.4	3.1	3.1	3.4	3.3	3.6	3.0	3.0	2.2	2.1
75	3.5	3.2	3.2	3.5	3.5	3.7	3.0	3.1	2.3	2.2
90	3.6	3.3	3.3	3.6	3.7	3.9	3.1	3.3	2.4	2.2
105	3.8	3.4	3.4	3.8	3.9	4.1	3.4	3.5	2.4	2.3
120	3.9	3.5	3.7	4.0	4.2	4.5			2.5	2.3
135	4.0	3.7	3.9	4.2			4.1	4.3	2.5	2.4
150	4.4	4.0	4.2	4.6	5.3	5.8	4.7	4.8	2.6	2.6
165	4.9	4.6	4.7	5.0	6.1	6.4			2.9	2.9
180	5.7	5.2			7.6	8.1	6.4	6.5	3.2	3.1
195	6.6	6.0	6.0	6.9	8.3	9.5	8.0	7.7	3.5	3.4

TABLE IV-2 -- Continued

Time (min)	Scale reading (index of torque) for water/cement ratio									
	0.40								0.42	
	Trial number									
	1	2	3	4	5	6	7	8	1	2
210	8.0	7.2	7.1	8.3	10.0	10.0	9.3	9.6	4.0	3.9
225	9.5	9.0	8.8	10.0	-	-	10.0	10.0	4.6	4.6
240	10.0	10.0	10.0	-			-	-	5.0	5.1
255									6.1	6.1
270									7.6	7.5
285									9.0	9.1
300									10.0	10.0
*)	238	233	236	225	210	208	214	213	295	290

*)The time when scale reading reached the value of 10.0

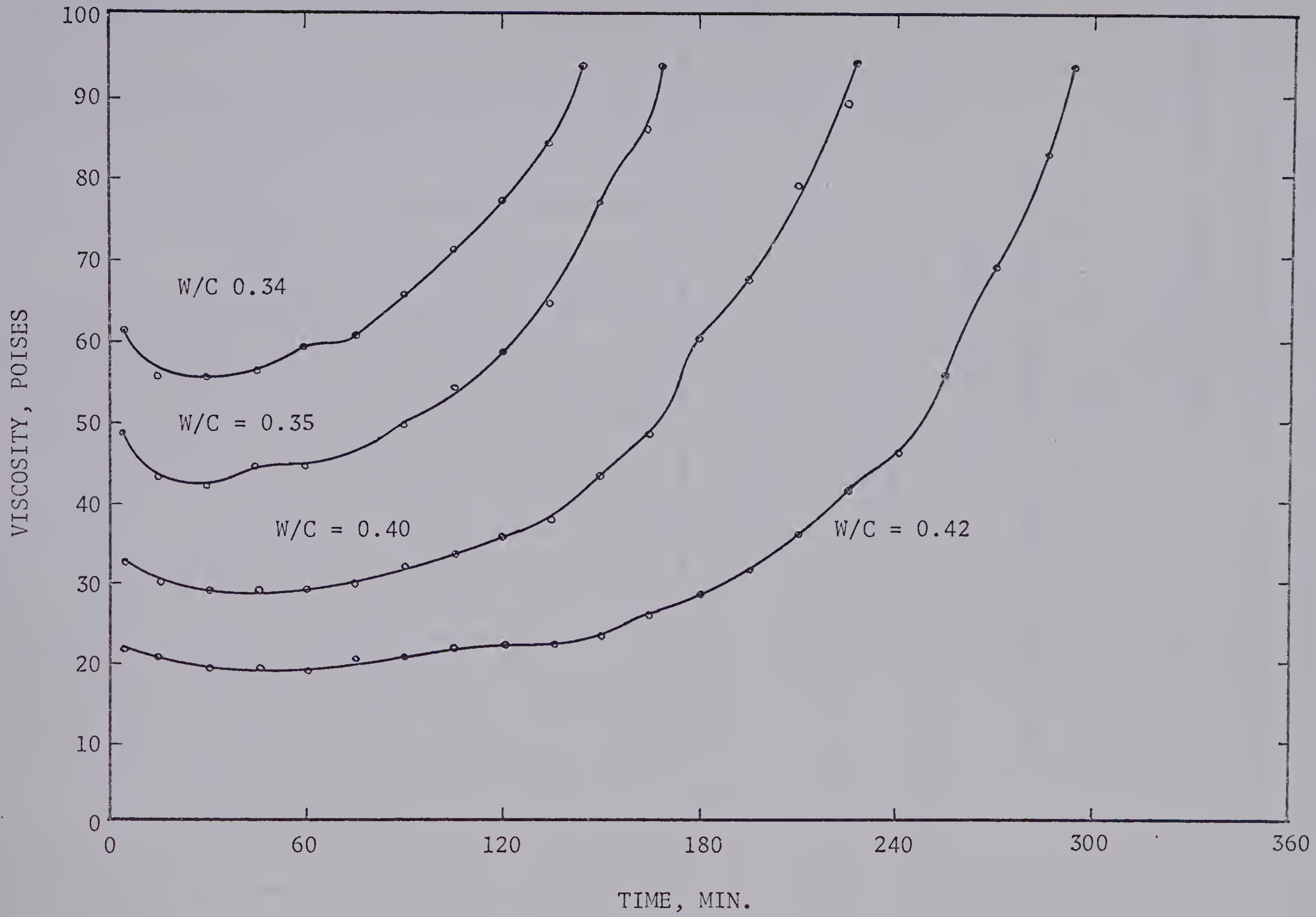

FIGURE IV.1 INLAND NORMAL PORTLAND CEMENT, WORKABILITY CURVES FOR DIFFERENT WATER/CEMENT RATIOS

4.1.1.2 Inland High Early Strength Cement

TABLES IV-3 and IV-4 show the variation of consistency with time for cement pastes of various water/cement ratio prepared from Inland High Early Strength Cement.

TABLE IV-3

CONSISTENCY IN UNITS OF "INDEX OF TORQUE" FOR

WATER/CEMENT RATIOS 0.40 AND 0.45

Time (min)	Scale reading (index of torque) for water/cement ratio							
	0.40		0.45					
	Trial number							
	1	2	1	2	3	4	5	6
5	8.5	8.3	7.2	7.2	7.0	6.9	7.3	7.4
15	7.7	7.6	6.0	6.0	6.5	6.5	6.6	6.8
30	7.6	7.5	6.1	5.8	6.3	6.2	6.2	6.4
45	7.7	7.8	5.9	5.8	6.4	6.3	6.4	6.4
60	8.2	8.2	6.0	6.0	6.6	6.5	6.5	6.6
75	8.9	8.8	6.3	6.4	7.0	6.9	6.9	7.0
90	9.2	9.5	7.0	7.0	7.3	7.1	7.1	7.5
105	10.0	10.0	7.5	7.6	8.2	7.9	7.8	8.1
120	-	-	8.2	8.2	8.6	8.3	8.3	8.6
135			9.2	9.2	9.2	8.9	9.1	9.2
150			9.5	10.0	10.0	9.6	10.0	10.0
165			10.0	-	-	10.0	-	-
*)	100	105	157	150	150	155	150	147

*)The time when scale reading reach the value of 10.0

TABLE IV-4

CONSISTENCY IN UNITS OF "INDEX OF TORQUE" FOR

WATER/CEMENT RATIOS 0.47 and 0.50

Time (min)	Scale reading (index of torque) for water/cement ratio							
	0.47		0.50					
	Trial number							
	1	2	1	2	3	4	5	6
5	5.2	5.2	3.9	3.8	4.0	3.9	4.0	3.9
15	4.7	4.9	3.5	3.4	3.6	3.5	3.5	3.6
30	4.5	4.9	3.5	3.4	3.6	3.5	3.6	3.6
45	4.6	5.0	3.5	3.5	3.7	3.6	3.6	3.7
60	4.8	5.1	3.5	3.6	3.9	3.8	3.8	3.9
75	5.2	5.5	3.8	3.8	3.9	3.9	4.0	4.0
90	5.6	5.9	4.0	3.9	4.1	4.1	4.2	4.2
105	6.0	6.2	4.2	4.2	4.5	4.4	4.5	4.4
120	6.5	6.9	4.5	4.5	4.9	4.8	4.8	4.9
135	7.0	7.1	4.9	4.9	5.3	5.2	5.2	5.3
150	7.5	7.8	5.2	5.2	6.0	5.7	5.8	6.0
165	8.1	8.5	5.9	6.1	7.4	7.1	7.0	7.1
180	9.1	9.3	6.9	7.1	9.2	9.1	8.3	8.7
195	10.0	10.0	8.1	8.5	10.0	10.0	10.0	10.0
210	-	-	10.0	10.0	-	-	-	-
*)	186	185	211	209	188	189	194	190

*)The time when scale reading reached the value of 10.0

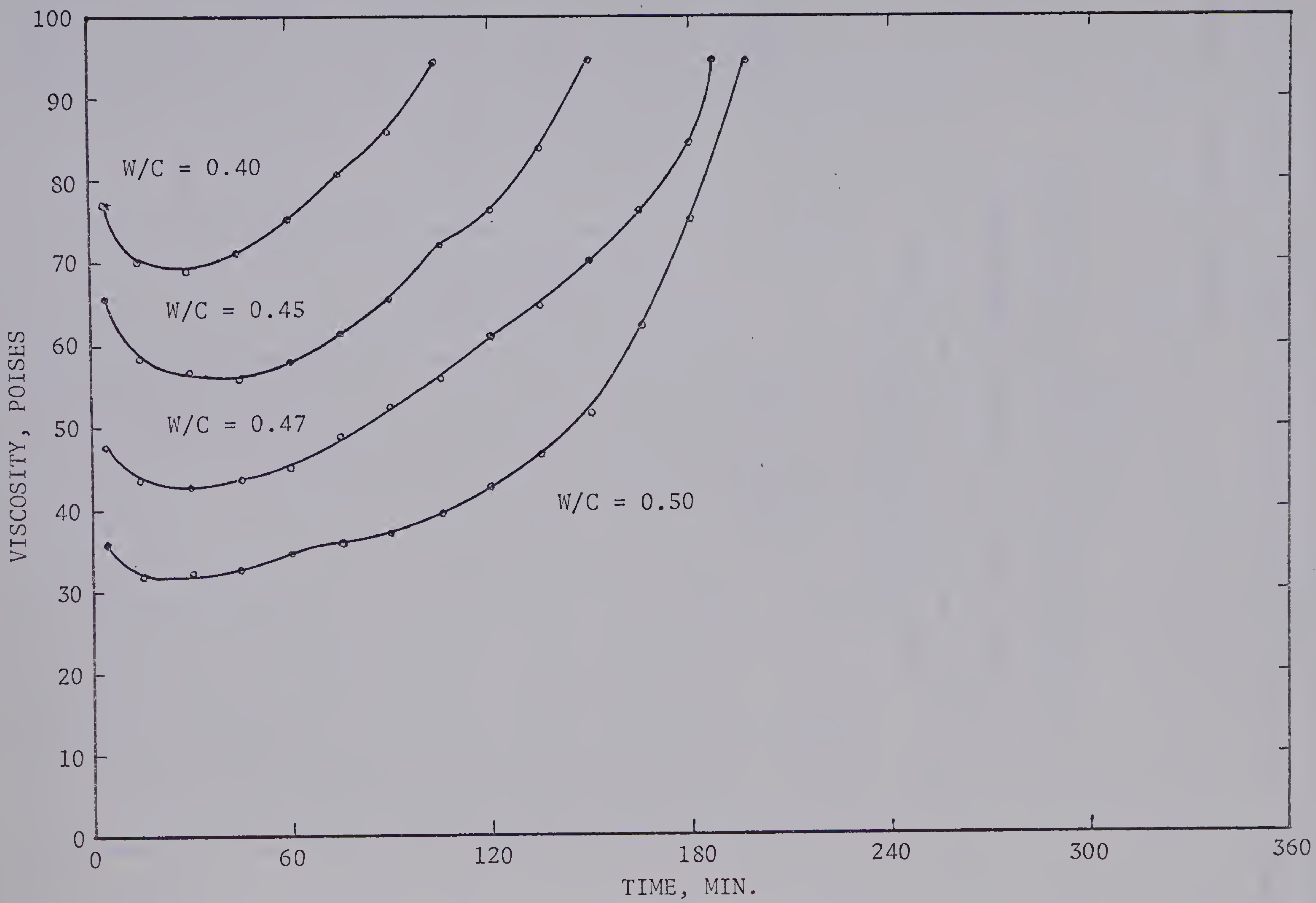

FIGURE IV.2 INLAND HIGH EARLY STRENGTH CEMENT, WORKABILITY CURVES FOR DIFFERENT WATER/CEMENT RATIOS

4.1.1.3 Inland Sulphate Resistant Cement

TABLES IV-5 and IV-6 show the variation of consistency with time for cement pastes of various water/cement ratio prepared from Inland Sulphate Resistant Cement.

TABLE IV-5

CONSISTENCY IN UNITS OF "INDEX OF TORQUE" FOR WATER/CEMENT RATIOS 0.30, 0.33 AND 0.34

Time (min)	Scale reading (index of torque) for water/cement ratio					
	0.30		0.33		0.34	
	Trial number					
	1	2	1	2	1	2
5	10.0	10.0	7.5	7.5	6.1	6.1
15	10.0	10.0	7.2	6.8	6.0	6.1
30	10.0	10.0	7.1	6.7	5.8	6.0
45	10.0	10.0	7.1	6.8	5.8	5.9
60	10.0	10.0	7.2	6.9	5.9	5.9
75	-	-	7.5	7.1	6.0	5.9
90			7.7	7.3	6.0	6.0
105			8.0	7.4	6.1	6.2
120			8.2	7.9	6.2	6.4
135			8.4	8.1	6.4	6.6
150			8.9	8.4	6.8	6.9
165			9.1	8.7	7.0	7.2

TABLE IV-5 -- Continued

Time (min)	Scale reading (index of torque) for water/cement ratio					
	0.30		0.33		0.34	
	Trial number					
	1	2	1	2	1	2
180	-	-	9.3	8.9	7.1	7.3
195			9.4	9.9	7.3	7.7
210			9.6	9.2	7.6	8.0
225			10.0	10.0	8.1	8.5
240			-	-	8.5	9.0
255					9.0	9.5
270					10.0	10.0
*)	-	-	216	225	281	266

*)The time when scale reading reached the value of 10.0

TABLE IV-6

CONSISTENCY IN UNITS OF "INDEX OF TORQUE" FOR

WATER/CEMENT RATIOS 0.35 AND 0.37

Time (min)	Scale reading (index of torque) for water/cement ratio						
	0.35						0.37
	Trial number						
	1	2	3	4	5	6	1
5	6.1	6.2	4.9	5.0	5.4	5.4	3.9
15	5.6	5.7	4.6	4.7	5.0	5.0	3.6
30	5.5	5.6	4.5	4.5	4.9	4.8	3.6
45	5.5	5.7	4.5	4.4	4.8	4.7	3.6
60	5.5	5.7	4.5	4.4	4.8	4.7	3.5
75	5.4	5.6	4.5	4.4	4.8	4.7	3.5
90	5.4	5.6	4.5	4.4	4.8	4.7	3.4
105	5.5	5.6	4.5	4.5	4.9	4.8	3.4
120	5.5	5.7	4.6	4.7	4.9	4.8	3.5
135	5.7	5.9	4.8	4.9	5.0	4.9	3.7
150	5.9	6.1	5.0	5.0	5.1	5.1	3.8
165	6.0	6.1	5.1	5.1	5.3	5.3	
180	6.3	6.4	5.2	5.2	5.5	5.5	3.9
195	6.4	6.5	5.4	5.5	5.7	5.7	4.0
210	6.8	6.9	5.6	5.7	5.9	5.9	4.2

TABLE IV-6 -- Continued

Time (min)	Scale reading (index of torque) for water/cement ratio						
	0.35						0.37
	Trial number						
	1	2	3	4	5	6	1
225	7.1	7.2	6.0	6.1	6.3	6.2	4.4
240	7.3	7.4	6.5	6.7	6.9	6.7	4.8
255	7.7	7.8	6.9	7.1	7.3	7.2	
270	8.5	8.6	7.8	7.9	8.0	7.9	5.3
285	9.3	9.5	8.3	8.7	8.5	8.3	5.8
300	10.0	10.0	9.0	9.5	9.1	9.1	6.2
315	-	-	9.5	10.0	9.7	10.0	6.7
330			10.0	-	10.0	-	7.3
345			-		-		7.9
360							8.6
375							9.1
390							9.8
405							10.0
*)	296	290	316	308	320	314	392

*)The time when scale reading reached the value of 10.0.

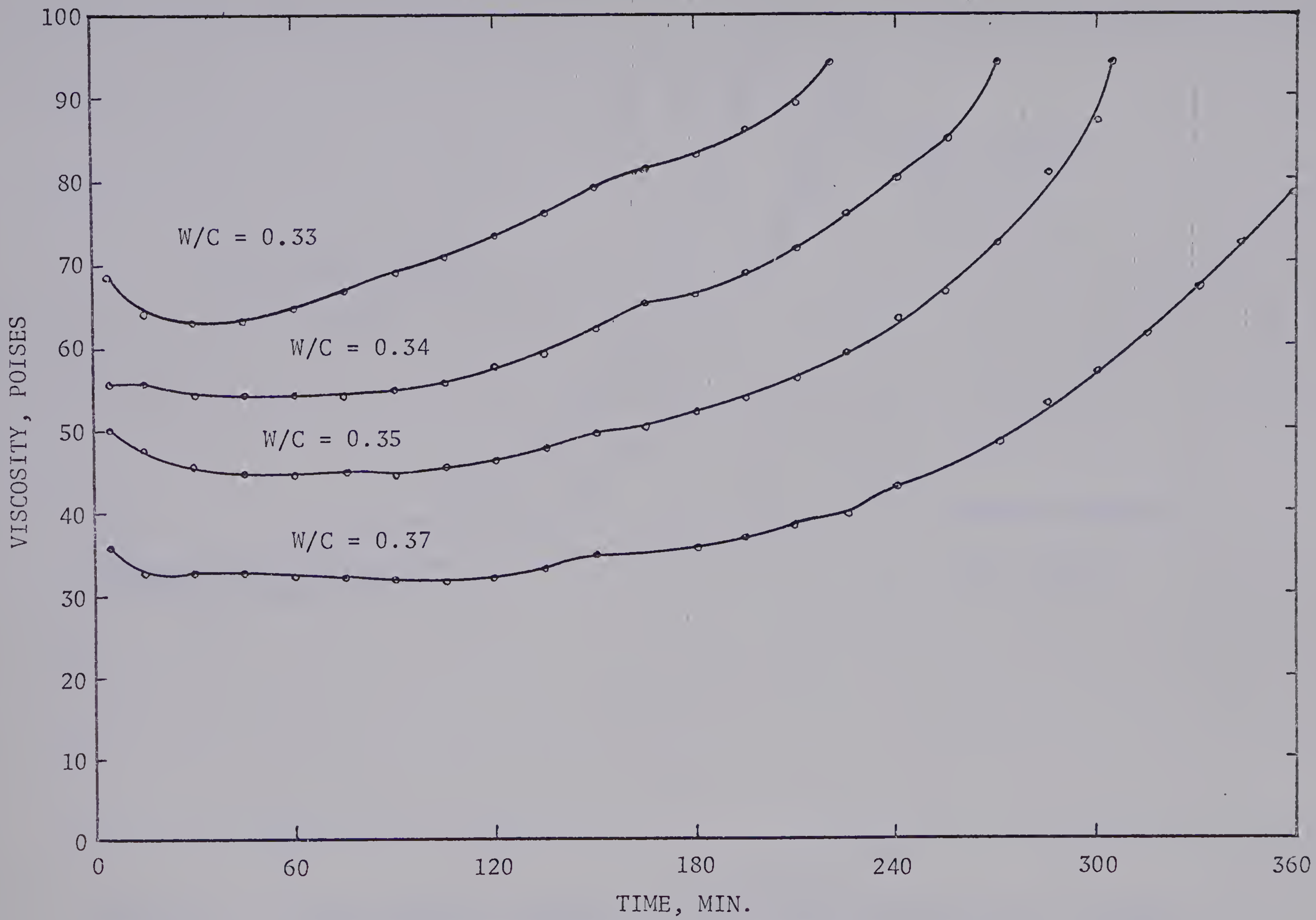

FIGURE IV.3 INLAND SULPHATE RESISTANT CEMENT, WORKABILITY CURVES FOR DIFFERENT WATER/CEMENT RATIOS

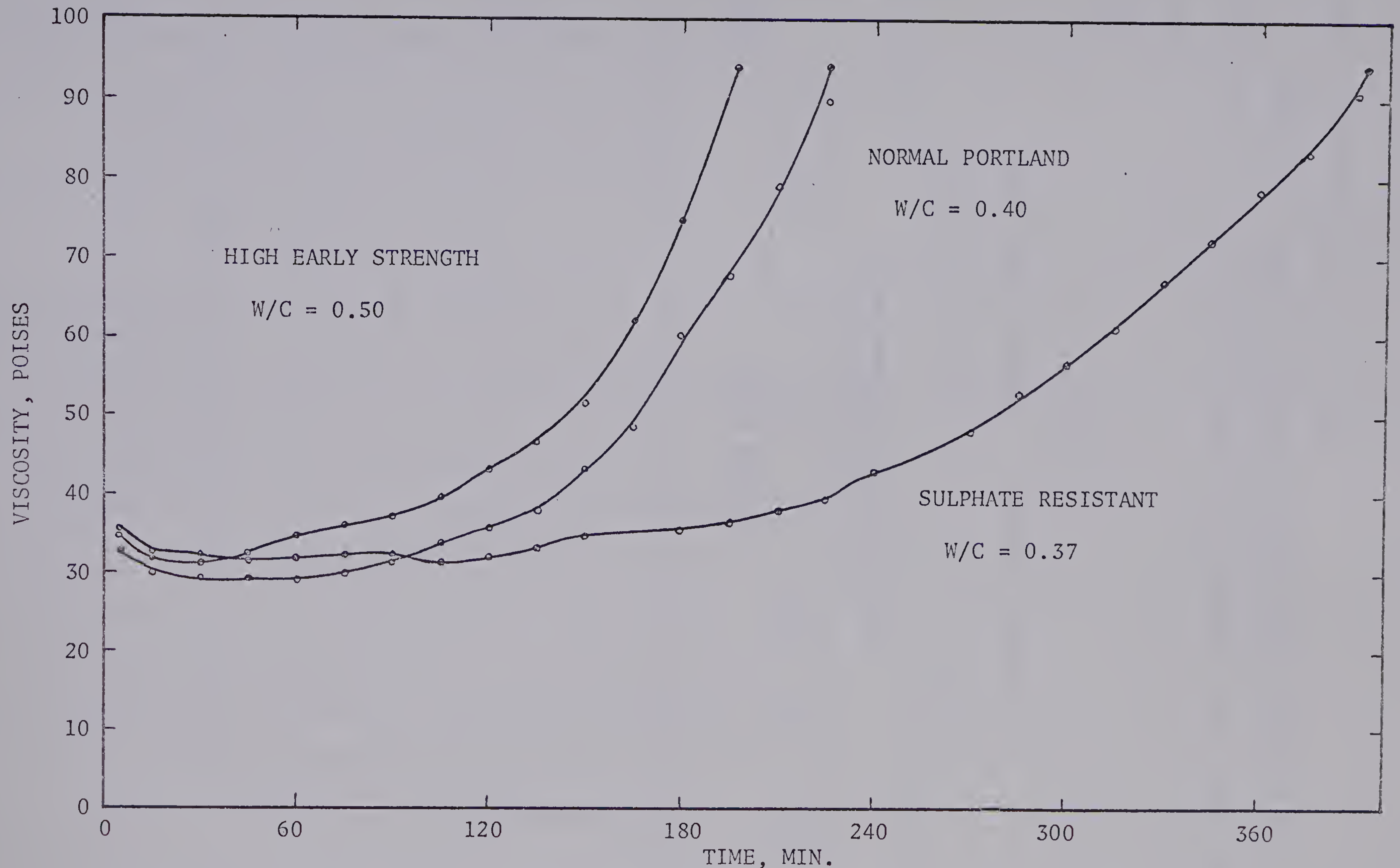

FIGURE IV.4 INLAND CEMENTS, WORKABILITY CURVES FOR DIFFERENT TYPES OF CEMENTS

4.1.2 Canada Normal Portland Cement

TABLE IV-7 shows the variation of consistency with time for cement pastes of various water/cement ratio prepared from Canada Normal Cement.

TABLE IV-7

CONSISTENCY IN UNITS OF "INDEX OF TORQUE" FOR WATER/CEMENT RATIOS 0.32, 0.33, 0.34 AND 0.35

Time (min)	Scale reading (index of torque) for water/cement ratio							
	0.32		0.33		0.34		0.35	
	Trial number							
	1	2	1	2	1	2	1	2
5	8.2	8.4	7.9	7.9	5.0	5.0	4.4	4.5
15	8.3	8.5	7.8	7.9	4.8	4.7	4.0	4.0
30	9.5	9.5	7.5	7.5	4.7	4.7	4.0	3.8
45	8.9	9.0	7.4	7.4	4.7	4.7	3.9	3.8
60	9.0	9.2	7.3	7.4	4.9	4.9	3.8	3.6
75	9.1	9.4	7.9	7.5	5.0	5.1	3.8	3.7
90	9.2	9.3	7.3	7.7	4.8	4.9	3.8	3.8
105	9.5	9.6	7.3	7.9	4.9	4.9	3.8	3.8
120	9.8	10.0	8.2	8.2	5.1	5.0	3.9	3.9
135	10.0	-	8.2	8.8	5.2	5.1	3.9	3.9
150	-		8.6	8.7	5.1	5.2	4.0	4.0
165			9.3	9.3	5.3	5.4	4.0	4.1
180			10.0	10.0	5.6	5.7	4.0	4.1

TABLE IV-7 -- Continued

Time (min)	Scale reading (index of torque) for water/cement ratio							
	0.32		0.33		0.34		0.35	
	Trial number							
	1	2	1	2	1	2	1	2
195			-	-	6.0	6.0	4.1	4.2
210					6.2	6.2	4.2	4.3
225					6.9	6.9	4.3	4.4
240					7.5	7.5	4.7	4.8
255					8.5	8.3	5.0	5.1
270					9.3	9.4	5.4	5.5
285					10.0	10.0	5.9	6.0
300					-	-	6.8	6.9
315							7.4	7.7
330							8.2	8.5
345							9.1	9.3
360							9.9	10.0
375							10.0	-
*)	130	126	175	178	285	280	363	355

*)The time when scale reading reached the value of 10.0.

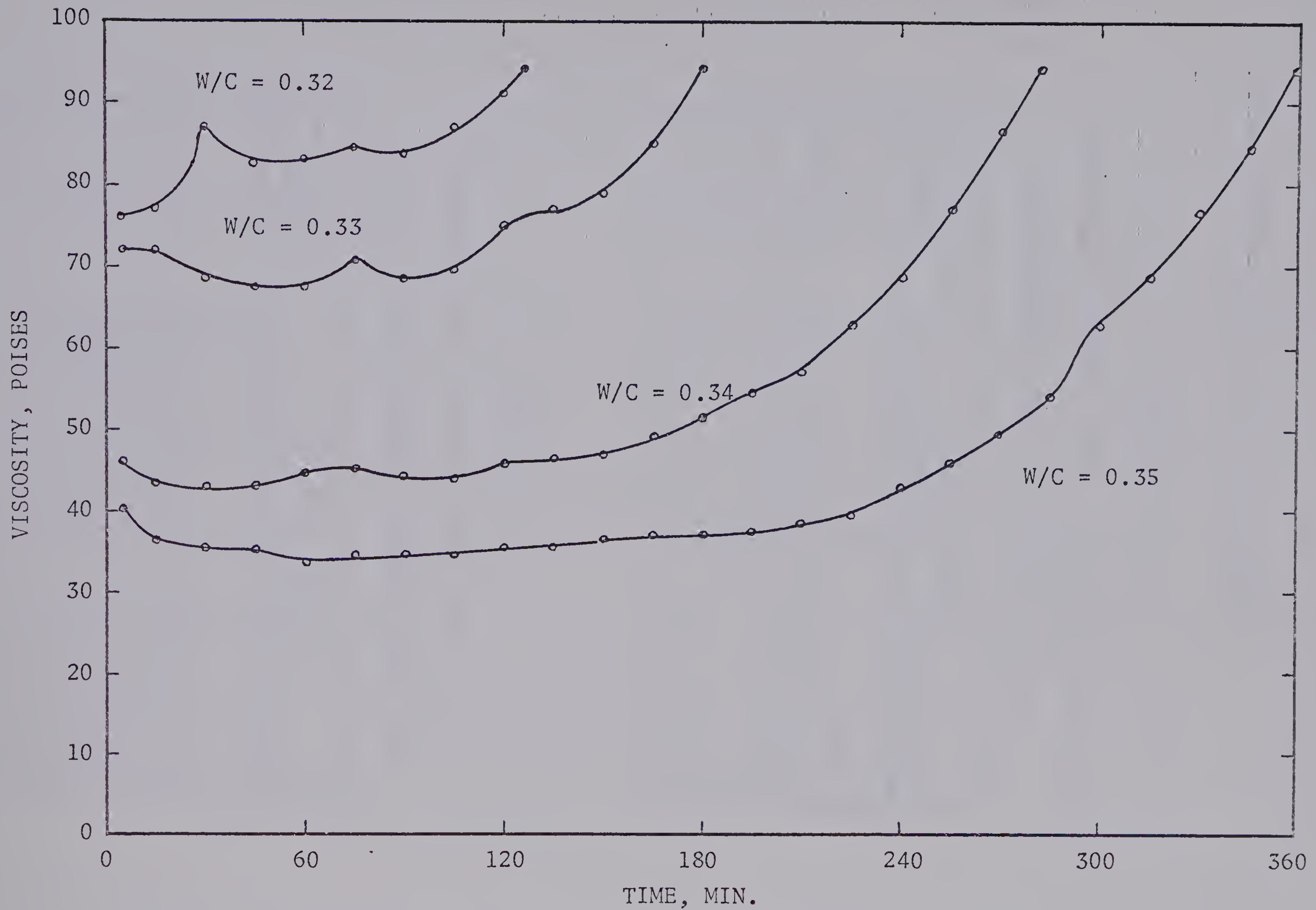

FIGURE IV.5 CANADA NORMAL PORTLAND CEMENT, WORKABILITY CURVES FOR DIFFERENT WATER/CEMENT RATIOS

4.2 Electrical Test

4.2.1 Results of Electrical and Vicat Tests Run Simultaneously with Workability Tests on Inland Cements:

TABLE IV-8

ELECTRICAL AND VICAT TESTS RUN ON THE INLAND HIGH EARLY STRENGTH CEMENT, ELECTRODE TYPE "AO"

Water/cement ratio	Setting time by: Vicat test	Electrical test
0.40		2 hrs. 38 min.
0.45	4 hrs. 30 min.	4 hrs. 11 min.
0.47	-	-
0.50	5 hrs. 20 min.	4 hrs. 21 min.

TABLE IV-9

ELECTRICAL AND VICAT TESTS RUN ON THE INLAND SULPHATE RESISTANT CEMENT, ELECTRODE TYPE "AO"

Water/cement ratio	Setting time by: Vicat test	Electrical test
0.33	5 hrs. 26 min.	5 hrs. 54 min.
0.34	5 hrs. 48 min.	5 hrs. 06 min.
0.35	6 hrs. 15 min.	5 hrs. 41 min.
0.37	5 hrs. 55 min.	4 hrs. 53 min.

4.2.2 Results of Electrical and Vicat Tests Run Simultaneously with Workability Tests on Canada Normal Portland Cement:

TABLE IV-10

ELECTRICAL AND VICAT TESTS RUN ON THE CANADA NORMAL PORTLAND CEMENT, ELECTRODE TYPE "AO"

Water/cement ratio	Setting time by: Vicat test	Electrical test
0.32	5 hrs. 20 min.	5 hrs. 20 min.
0.33	5 hrs. 19 min.	5 hrs. 06 min.
0.34	5 hrs. 34 min.	5 hrs. 20 min.
0.35	5 hrs. 35 min.	5 hrs. 50 min.

4.2.3 Results of Electrical and Vicat Tests Run on Canada Normal Portland Cement Pastes of Normal Consistency Using Various Types of Electrodes:

TABLE IV-11

"AC" TYPE ELECTRODE, w/c = 0.28

Test No.	Setting time by: Vicat test	Electrical test
AC 1	4 hrs. 55 min.	4 hrs. 30 min.
AC 2	4 hrs. 18 min.	4 hrs. 46 min.
AC 3	4 hrs. 32 min.	2 hrs. 46 min.*
AC 4	4 hrs. 41 min.	5 hrs. 28 min.
AC 5	4 hrs. 29 min.	3 hrs. 34 min.*
AC 6	4 hrs. 33 min.	3 hrs. 10 min.*
AC 7	4 hrs. 27 min.	5 hrs. 16 min.
AC 8	4 hrs. 36 min.	4 hrs. 31 min.
AC 9	4 hrs. 50 min.	5 hrs. 22 min.
AC 10	4 hrs. 05 min.	1 hr. 08 min.**
AC 11	4 hrs. 15 min.	4 hrs. 27 min.*

*There was more than one characteristic on the electrical record indicating setting time.

**Observation inconsistent with other data.

TABLE IV-12

"AO" TYPE ELECTRODE, w/c = 0.28

Test No.	Setting time by: Vicat test	Electrical test
AO 1	4 hrs. 45 min.	4 hrs. 38 min.
AO 2	4 hrs. 50 min.	4 hrs. 40 min.
AO 3	4 hrs. 48 min.	4 hrs. 43 min.
AO 4	4 hrs. 37 min.	5 hrs. 36 min.
AO 5	4 hrs. 46 min.	4 hrs. 52 min.
AO 6	4 hrs. 37 min.	5 hrs. 06 min.
AO 7	4 hrs. 26 min.	4 hrs. 25 min.
AO 8	4 hrs. 19 min.	4 hrs. 31 min.
AO 9	4 hrs. 39 min.	4 hrs. 50 min.
AO 10	4 hrs. 36 min.	5 hrs. 07 min.
AO 11	4 hrs. 45 min.	4 hrs. 33 min.
AO 12	4 hrs. 12 min.	4 hrs. 30 min.
AO 13	4 hrs. 21 min.	4 hrs. 36 min.
AO 14	4 hrs. 30 min.	5 hrs. 05 min.
AO 15	4 hrs. 30 min.	4 hrs. 53 min.
AO 16	4 hrs. 20 min.	4 hrs. 49 min.
AO 17	4 hrs. 25 min.	4 hrs. 36 min.
AO 18	4 hrs. 31 min.	4 hrs. 54 min.
AO 19	5 hrs. 03 min.	5 hrs. 18 min.
AO 20	4 hrs. 35 min.	5 hrs. 05 min.
AO 21	4 hrs. 10 min.	5 hrs. 12 min.

TABLE IV-13

"BC" TYPE ELECTRODE, w/c = 0.28

Test No.	Setting time by: Vicat test	Electrical test
BC 1	4 hrs. 32 min.	4 hrs. 37 min.
BC 2	4 hrs. 38 min.	5 hrs. 11 min.
BC 3	4 hrs. 27 min.	5 hrs. 05 min.
BC 4	4 hrs. 43 min.	4 hrs. 16 min.
BC 5	4 hrs. 27 min.	4 hrs. 36 min.
BC 6	4 hrs. 30 min.	4 hrs. 48 min.
BC 7	4 hrs. 03 min.	4 hrs. 46 min.
BC 8	4 hrs. 48 min.	4 hrs. 43 min.
BC 9	4 hrs. 32 min.	4 hrs. 39 min.
BC 10	4 hrs. 36 min.	5 hrs. 48 min.
BC 11	4 hrs. 39 min.	4 hrs. 46 min.

TABLE IV-14

"BO" TYPE ELECTRODE, w/c = 0.28

Test No.	Setting time by: Vicat test	Electrical test
BO 1	4 hrs. 06 min.	4 hrs. 28 min.
BO 2	3 hrs. 55 min.	3 hrs. 18 min.
BO 3	4 hrs. 13 min.	4 hrs. 01 min.
BO 4	4 hrs. 00 min.	4 hrs. 22 min.
BO 5	4 hrs. 18 min.	4 hrs. 16 min.
BO 6	4 hrs. 21 min.	4 hrs. 31 min.
BO 7	4 hrs. 33 min.	4 hrs. 06 min.
BO 8	4 hrs. 18 min.	3 hrs. 49 min.
BO 9	4 hrs. 32 min.	2 hrs. 58 min.*
BO 10	4 hrs. 37 min.	4 hrs. 25 min.
BO 11	4 hrs. 24 min.	4 hrs. 20 min.

*There was more than one characteristic on the electrical record indicating setting time.

TABLE IV-15

"C" TYPE ELECTRODE, w/c = 0.28

Test No.	Setting time by: Vicat test	Electrical test
C 1	4 hrs. 45 min.	4 hrs. 48 min.
C 2	4 hrs. 10 min.	7 hrs. 12 min.
C 3	4 hrs. 24 min.	4 hrs. 48 min.
C 4	4 hrs. 27 min.	4 hrs. 30 min.
C 5	4 hrs. 40 min.	5 hrs. 06 min.

4.2.4 Results of Electrical and Vicat Tests Run on Canada Normal Portland Cement Pastes with a Higher Water Content than the Normal Consistency Value:

TABLE IV-16

"AO" TYPE ELECTRODE, w/c = 0.357

Test No.	Setting time by: Vicat test	Setting time by: Electrical test
101	5 hrs. 05 min.	5 hrs. 46 min.
102	6 hrs. 05 min.	6 hrs. 24 min.
103	6 hrs. 10 min.	5 hrs. 30 min.
104	6 hrs. 00 min.	5 hrs. 45 min.
105	6 hrs. 00 min.	5 hrs. 42 min.

4.2.5 Results of Electrical and Penetration Resistance Tests on Cement Mortars:

TABLE IV-17

"AO" TYPE ELECTRODE

Test No.	Setting time by: Penetration resistance test	Electrical test
M 1		*
M 2		*
M 3	5 hrs. 05 min.	4 hrs. 28 min.
M 4	5 hrs. 45 min.	4 hrs. 39 min.
M 5	5 hrs. 05 min.	4 hrs. 05 min.
M 6	5 hrs. 20 min.	*
M 7	5 hrs. 50 min.	4 hrs. 51 min.
M 8	5 hrs. 30 min.	*
M 9	5 hrs. 05 min.	4 hrs. 21 min.
M 10	4 hrs. 55 min.	4 hrs. 59 min.
M 11	5 hrs. 15 min.	*
M 12	5 hrs. 00 min.	4 hrs. 15 min.
M 13	5 hrs. 20 min.	4 hrs. 27 min.
M 14	5 hrs. 25 min.	*
M 15	5 hrs. 40 min.	*
M 16	5 hrs. 05 min.	*

*Electrical test record could not be evaluated.

Mix data concerning TABLE IV-17 tests:

Cement: Canada Normal Portland Cement

Aggregate: Standard Sand 20-30

Cement/aggregate ratio 0.333

Water/(cement + aggregate) ratio 0.10575

CHAPTER V

EVALUATION AND DISCUSSION OF THE RESULTS

5.1 Evaluation and Discussion of the Workability Test Results

5.1.1 Inland Cements

All tests on the three types of Inland cements resulted in a decrease in consistency - increase in workability during the first stage of the test. This probably corresponds to the so-called "dormant" period in the hydration of the cement paste (Troxell, Davis and Kelly, 1968).

In order to be within the viscosity range of the test equipment a water/cement ratio of 0.34 to 0.42 was required for the Inland normal Portland cements (FIGURE IV.1). This water content resulted in viscosities from 21.5 to 61 poises at the start of the test.

The increase in workability lasted approximately 30 minutes for the water/cement ratios 0.34 and 0.35 and 60 minutes for water/cement ratios 0.40 and 0.42. The specimen with water/cement ratio of 0.42 indicated a period of constant workability ranging from 105 to 135 minutes in length. After the increase there was a decrease in workability with time for all specimens.

Inland High Early Strength cement (FIGURE IV.2) showed a short period of workability increase at the start of the tests. Water/cement

ratios 0.40; 0.45; 0.47 and 0.50 resulted in initial viscosities of 77, 65.5, 47.5 and 35.5 poises. There was no further increase in the workability after this period. Rate of workability decrease during the latter period of all tested samples was variable and this difference is listed in TABLE V-1 as viscosity increase in poises during the last thirty minutes of test.

TABLE V-1

RATE OF VISCOSITY INCREASE OF INLAND HIGH EARLY STRENGTH CEMENT PASTES

Water/cement ratio	Viscosity increase during last 30 minutes of test, poises
0.40	14.0
0.45	21.5
0.47	28.5
0.50	36.5

Inland Sulphate Resistant Cement paste (FIGURE IV.3) appeared to be the most sensitive to water content of the three types of Inland cements tested. Water/cement ratios of 0.33, 0.34, 0.35 and 0.37 resulted in a range of viscosity between 68.5 and 35.5 poises at the start of the tests. This type of cement showed comparatively long periods of time, 30 to 105 minutes from the start of the tests, when workability of the cement pastes was increasing. The length of this period appeared to be a function of the mix water content.

When the three types of Inland cements having a similar initial

value of viscosity are compared (FIGURE IV.4), the workability curves of the High Early Strength Cement (water/cement ratio 0.50) and the Normal Portland Cement (water/cement ratio 0.40) are very similar; the Sulphate Resistant Cement, though of the lowest water content (water/cement ratio 0.37) reached the viscosity limit of the testing equipment 200 minutes later than the High Early Strength Cement and 166 minutes later than the Normal Portland Cement.

5.1.2 Canada Normal Portland Cement

Canada Normal Portland Cement appeared to be very sensitive to water content. Tested water/cement ratios of 0.32, 0.33, 0.34 and 0.35 resulted in initial viscosities of 76, 72, 46 and 40.5 poises (FIGURE IV.5). There was a considerably longer period of time, relative to Inland cements, varying from 90 to 180 minutes when the workability either increased or remained constant.

When the results of the Inland and the Canada Normal Portland cements are compared (FIGURE IV.1 and FIGURE IV.5) the testing time of the latter is much longer. The difference between the time when the Canada Normal Portland Cement (water/cement ratio 0.33) and the Inland Normal Portland Cement (water/cement ratio 0.35) reached the upper viscosity limit of the testing apparatus was 108 minutes, in spite of the similar initial viscosity values and the fact that both were normal cements.

All Canada Normal Portland Cement pastes were simultaneously tested by the electrical and the workability tests. The results are compared on FIGURES V.1, V.2, V.3 and V.4. A comparison of the results

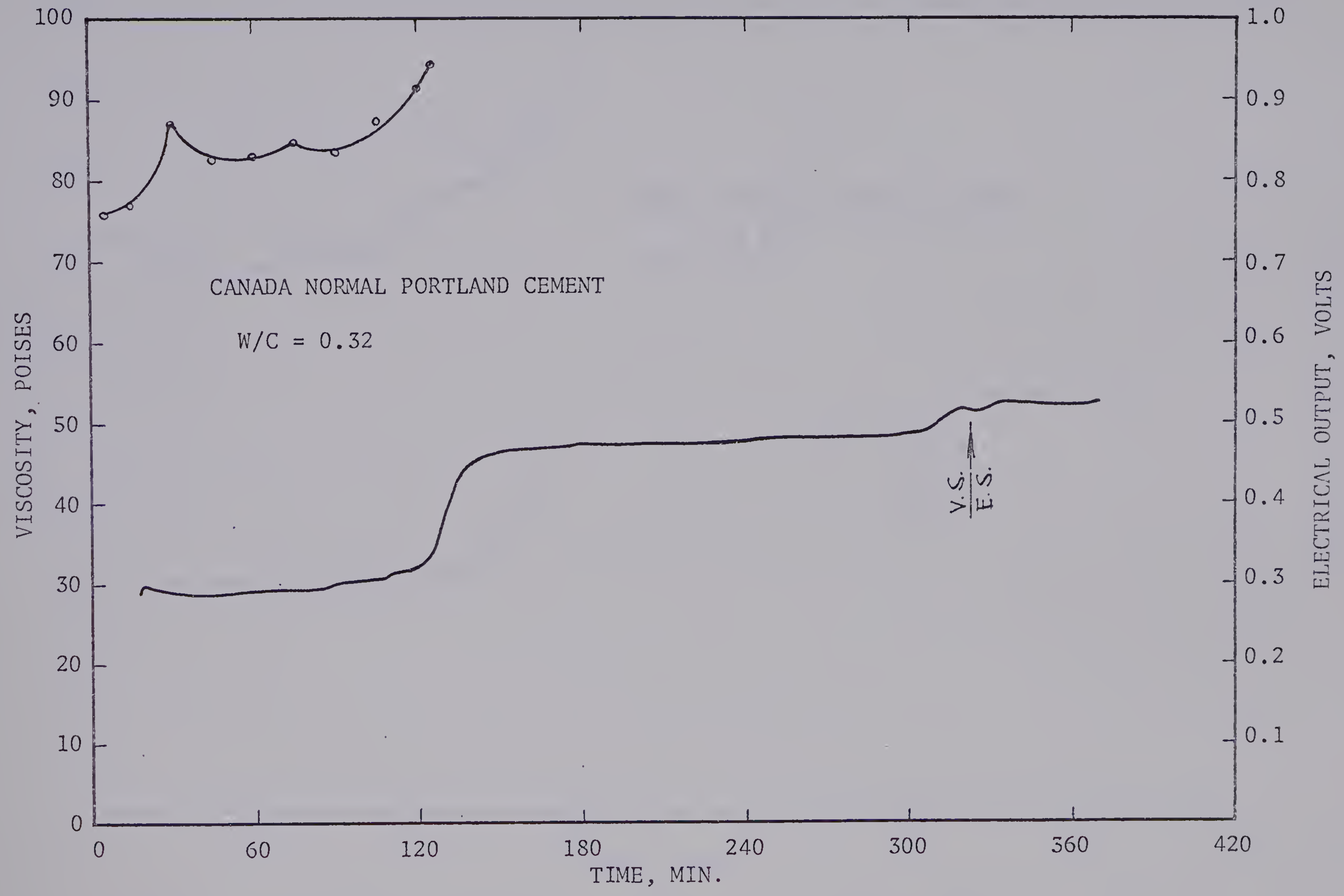

FIGURE V.1 COMPARISON OF WORKABILITY AND ELECTRICAL TESTS RESULTS

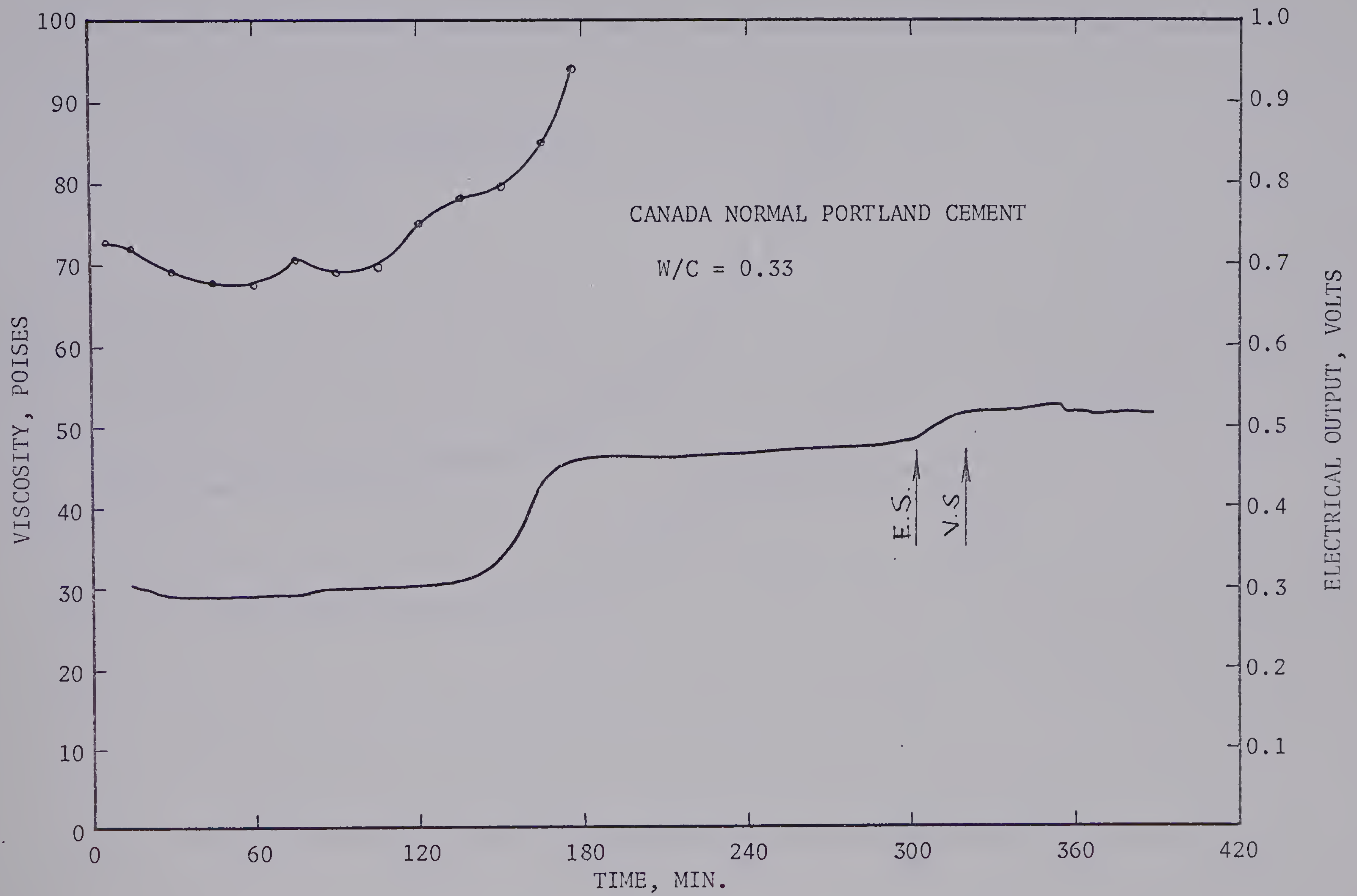

FIGURE V.2 COMPARISON OF WORKABILITY AND ELECTRICAL TESTS RESULTS

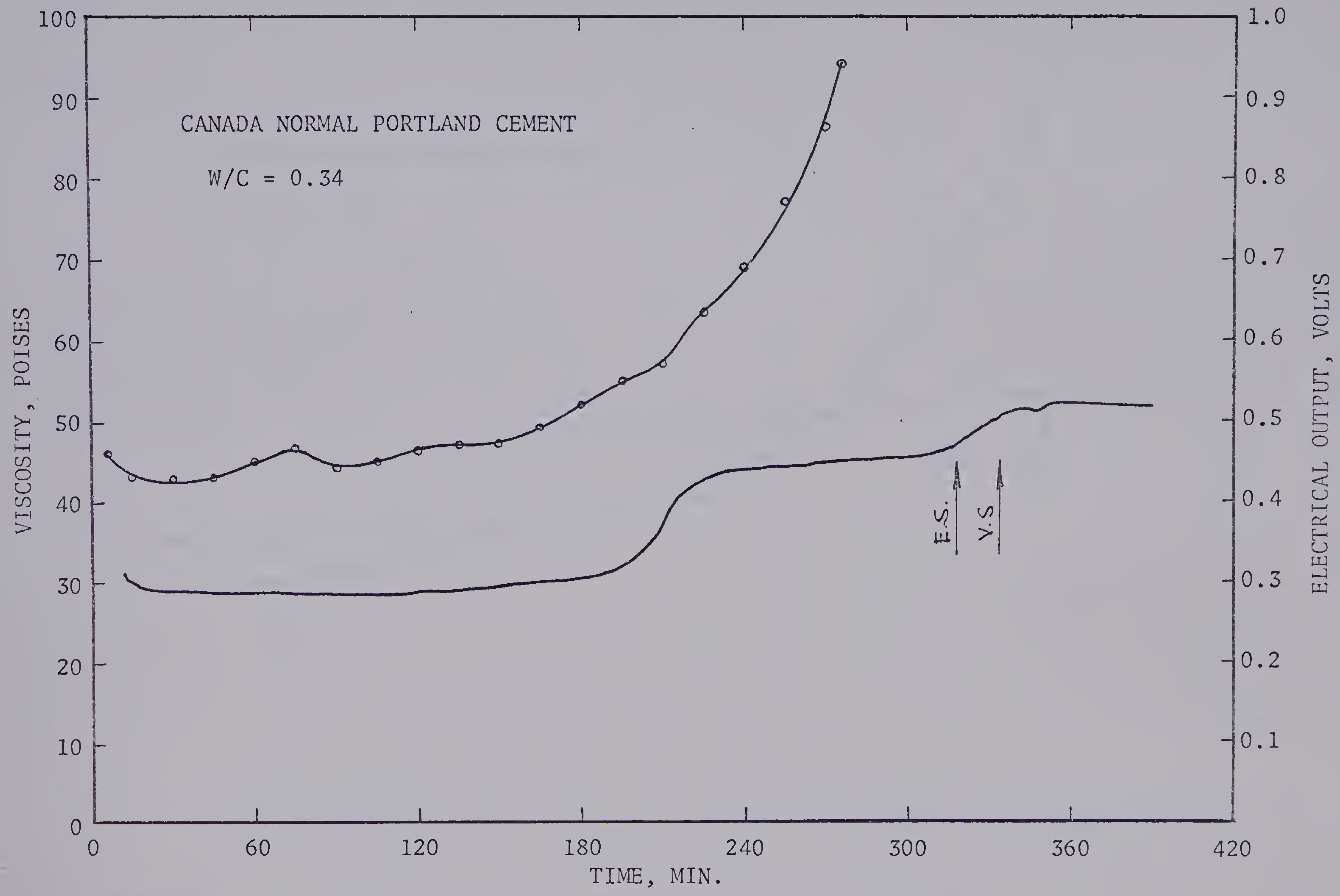

FIGURE V.3 COMPARISON OF WORKABILITY AND ELECTRICAL TEST RESULTS

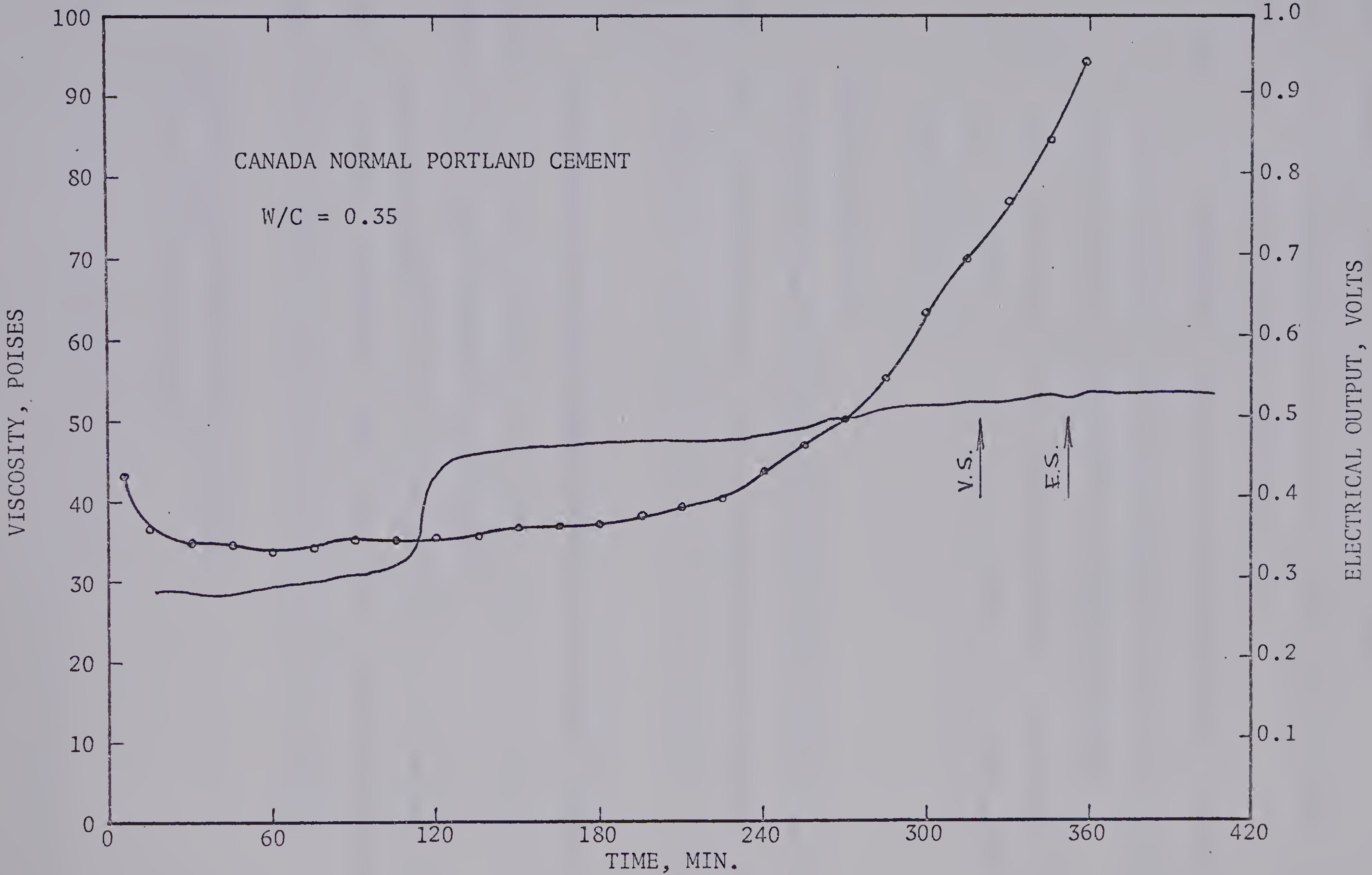

FIGURE V.4 COMPARISON OF WORKABILITY AND ELECTRICAL TESTS RESULTS

of the workability test and the electrical test does not appear to yield any significant relationship on the basis of data here presented. This may be for a number of reasons among which are:

(a) in the workability test the paste is continuously in motion and therefore subject to mechanical abrasion and in the electrical test the paste is at rest and can develop structure,

(b) appreciable water loss (bleeding) occurred in the electrical test as opposed to none in the workability test during the observation period.

5.1.3 General Discussion

The results above show that Inland High Early Strength and Inland Normal Portland cements have a shorter workability time and that they are less sensitive to water content than the Inland Sulphate Resistant Cement.

Canada Normal Portland Cement indicates an erratic tendency with respect to workability during the interval from start of test to the period of rapid final decrease in workability.

Continuing hydration of all cements results in decreased workability (increased consistency) until finally the internal friction of cement pastes reaches the upper limit of the apparatus capacity.

5.2 Evaluation and Discussion of the Vicat and Electrical Tests Results

5.2.1 Evaluation of Electrical Test Results

The voltage-time curves obtained from this work followed one of

three typical patterns as shown in FIGURE V.5.

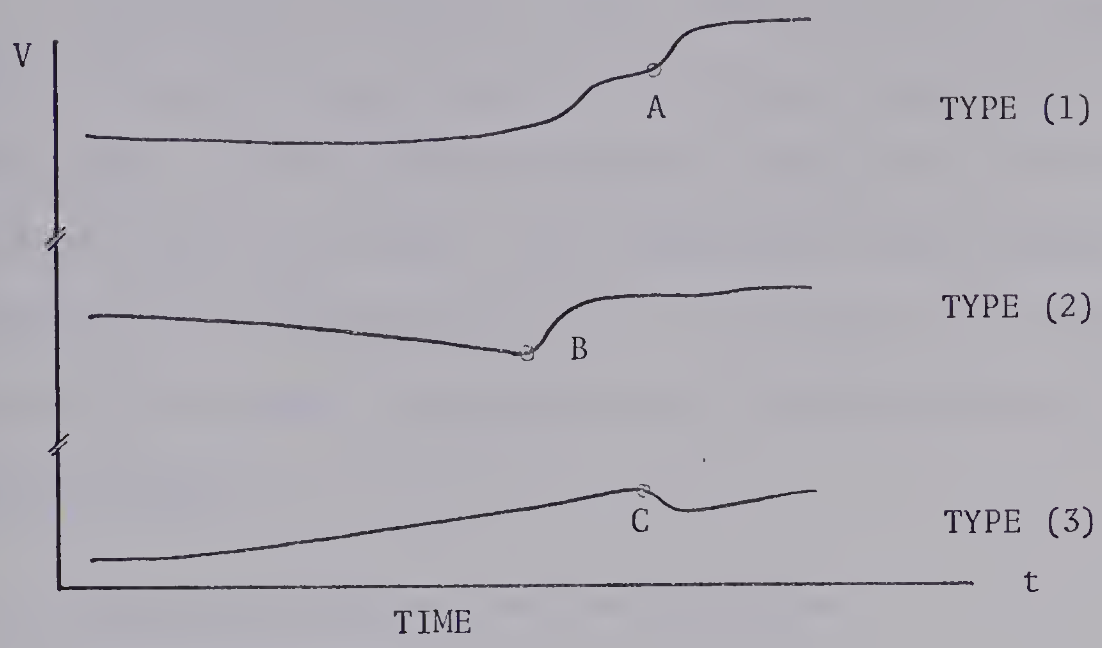

FIGURE V.5 THREE TYPICAL PATTERNS OF VOLTAGE-TIME CURVES

Based upon the appropriate figure the setting time can be determined in the following manner:

TYPE (1) - at base of second steepest positive $\frac{dV}{dt}$ incline (POINT A),

TYPE (2) - at base of first positive $\frac{dV}{dt}$ which occurs after a general negative $\frac{dV}{dt}$ (POINT B),

TYPE (3) - at beginning of first negative $\frac{dV}{dt}$ which occurs after a general positive $\frac{dV}{dt}$ (POINT C).

The above rules are based on an analysis of the electrical test and Vicat test results. Three typical voltage-time curves that were obtained during testing are shown in FIGURES V.6, V.7 and V.8. The changes which the above three rules deal with are relatively small and it would be necessary to use a recording voltmeter that permits extension

of its testing range to amplify the above mentioned changes.

It should be noted here, that cements tested in Czechoslovakia three or four years ago resulted in different characteristic voltage-time curves. That different character enabled one to determine the "setting time" more easily. The voltage-time curve patterns shown in FIGURES V.6, V.7 and V8 are similar to those that were obtained during testing fly ash-quick lime-gypsum mixes in 1967 and 1968 in Czechoslovakia.

5.2.2 Statistical Theory Used for Evaluation of the Tests Results.

The Mann-Whitney (Wilcoxon) W test for two samples was used (Guttman and Wilks, 1965).

Suppose that x_i $(i = 1,2, \ldots, m)$ and x_j' $(j = 1,2, \ldots, n)$ are independent samples from populations having continuous c.d.f.'s (continuous differential functions) $F_1(x)$ and $F_2(x)$. Pool both kinds of results together into a simple sample of $m + n$ observations and let the order statistics of this sample be y_k, where $k = 1,2, \ldots, m + n$. Let ranks R of all y's represent the elements of x_i and consider the sum of these ranks be T and consider W as a random variable defined as follows:

$$W = mn + \frac{m(n + 1)}{2} - T \qquad [3]$$

If both samples come from populations having identical c.d.f.'s, then for m and n both larger than 8, W has for all practical purposes

approximately a normal distribution with mean

$$E(W) = \frac{mn}{2}\ , \qquad [4]$$

and variance

$$Var(W) = \frac{mn(m + n + 1)}{12}\ . \qquad [5]$$

Rule for rejection of the null hypothesis:

$H_0 : F_1(x) \equiv F_2(x)$ is

$$\left| \frac{W - E(W)}{\sqrt{Var(W)}} \right| > Z_{\alpha/2} \qquad [6]$$

The 5 per cent level of significance was chosen for the evaluation of results,

hence: $\alpha = 0.05$ and

$Z_{\alpha/2} = 1.96$ (tabulated value).

Since the above Wilcoxon W Test does not allow smaller sample size than 8 for m or n, the method of paired comparisons was used for all sets of results having population of 5. This method tests the hypothesis

$H_0 : h = 0$

against alternatives

$H_1 : h \neq 0$

Sample of n independent pairs of experiments which produce pairs

of random variables y_{i1} and y_{i2} was performed. It was assumed that differences $y_{i2} - y_{i1} = x_i$ where $i = (1,2, \ldots, n)$, were independent random variables having identical normal distribution, with $\bar{x}$ and s^2 as the mean and the variance of the sample of x_i's.

The critical rule for this method is given by inequality

$$\left| \frac{\bar{x}\sqrt{n}}{s} \right| > t_{n-1;\alpha/2} \qquad [7]$$

where $t_{n-1;\alpha/2}$ is a tabulated value.

The 5 per cent level of significance was considered.

5.2.3 Evaluation of Results

TABLES V-1, V-2, V-3, V-4, V-5, V-6 and V-7 concern the Canada Normal Portland cement pastes result for various types of electrodes. Water content for all tested cement pastes was determined by the Normal Consistency procedure unless otherwise mentioned in the titles of tables.

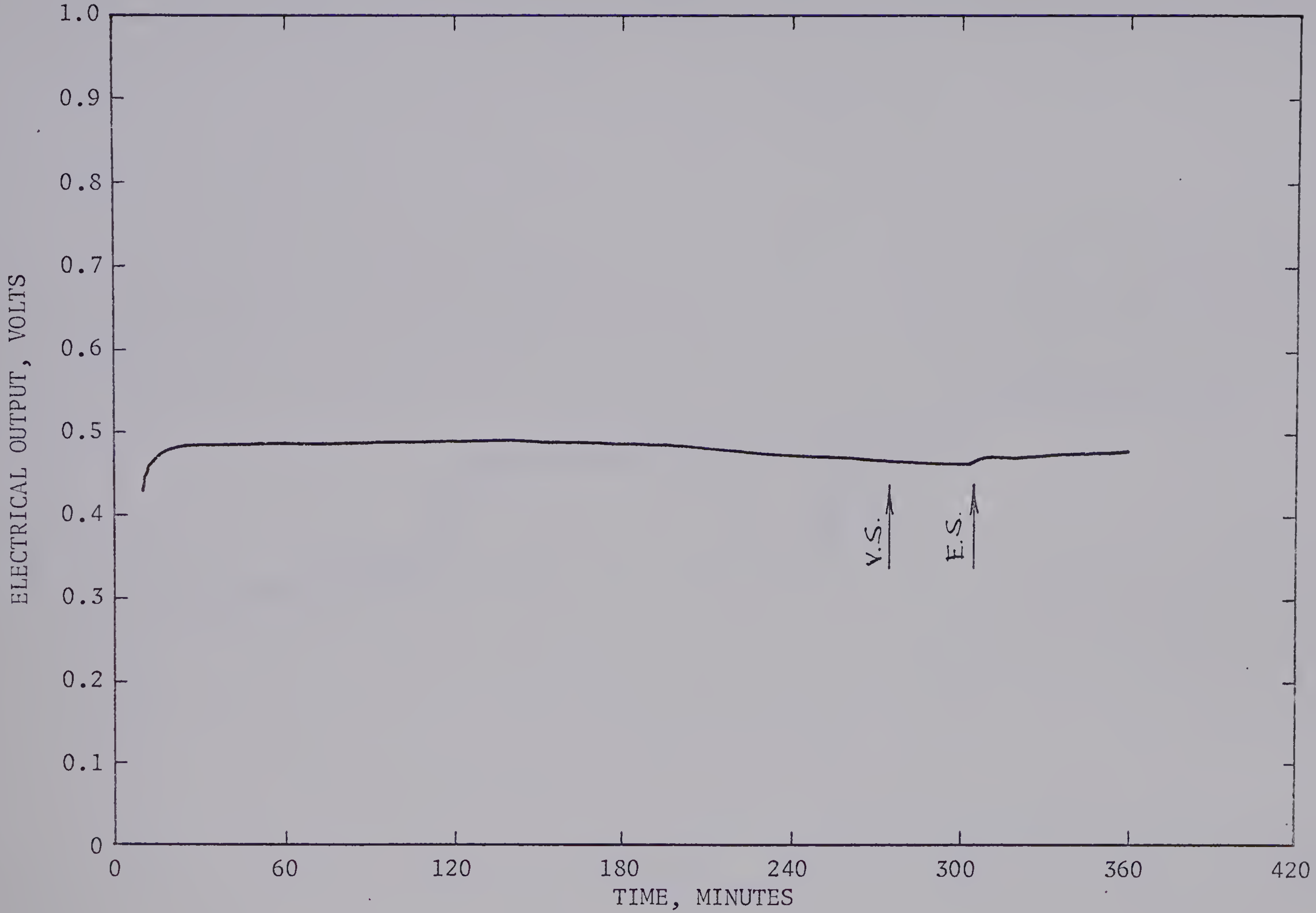

FIGURE V.6 TYPICAL RECORD OF ELECTRICAL TEST; TEST NO. AO 20, TYPE (2)

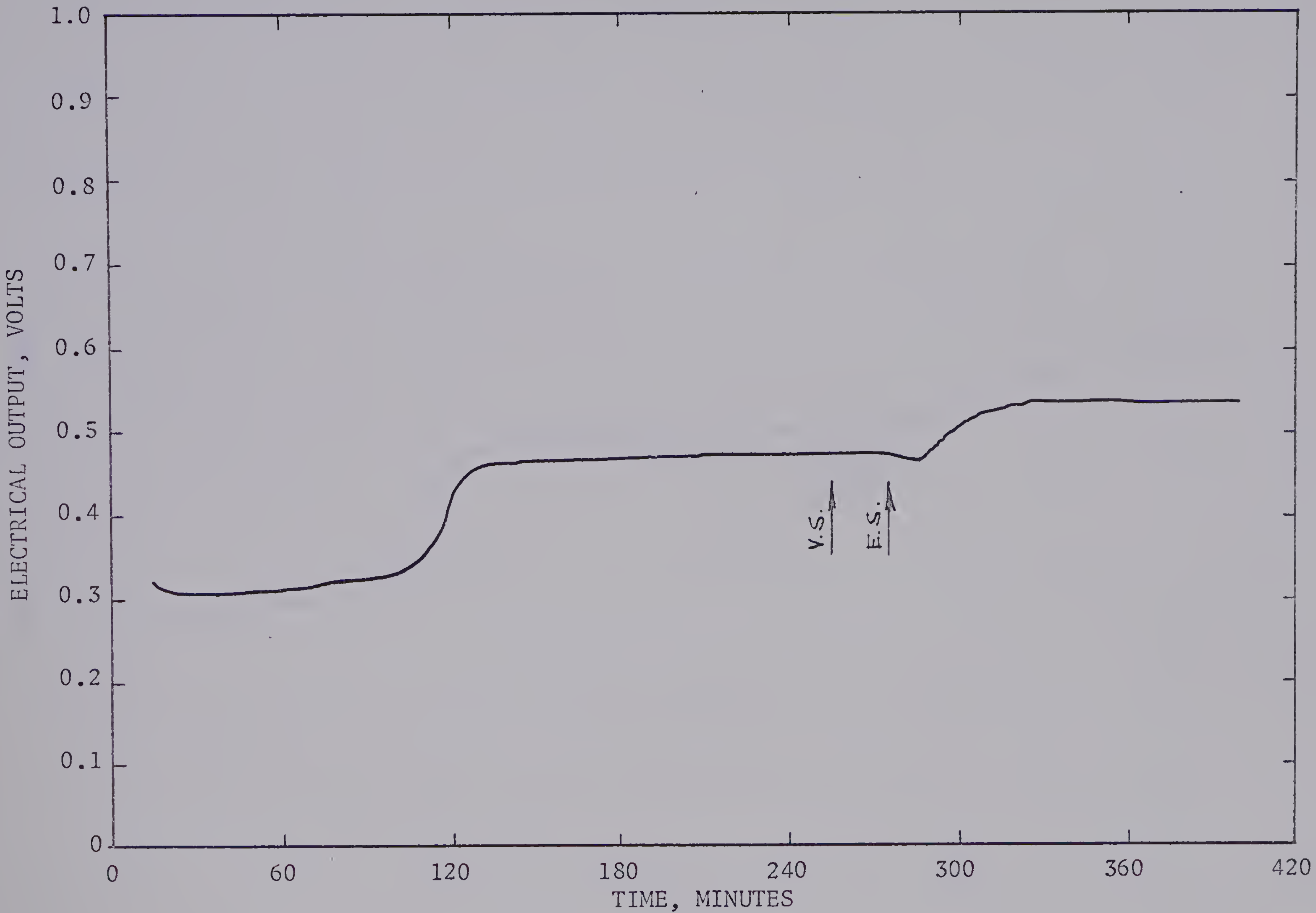

FIGURE V.7 TYPICAL RECORD OF ELECTRICAL TEST; TEST NO. AO 17 TYPE (3)

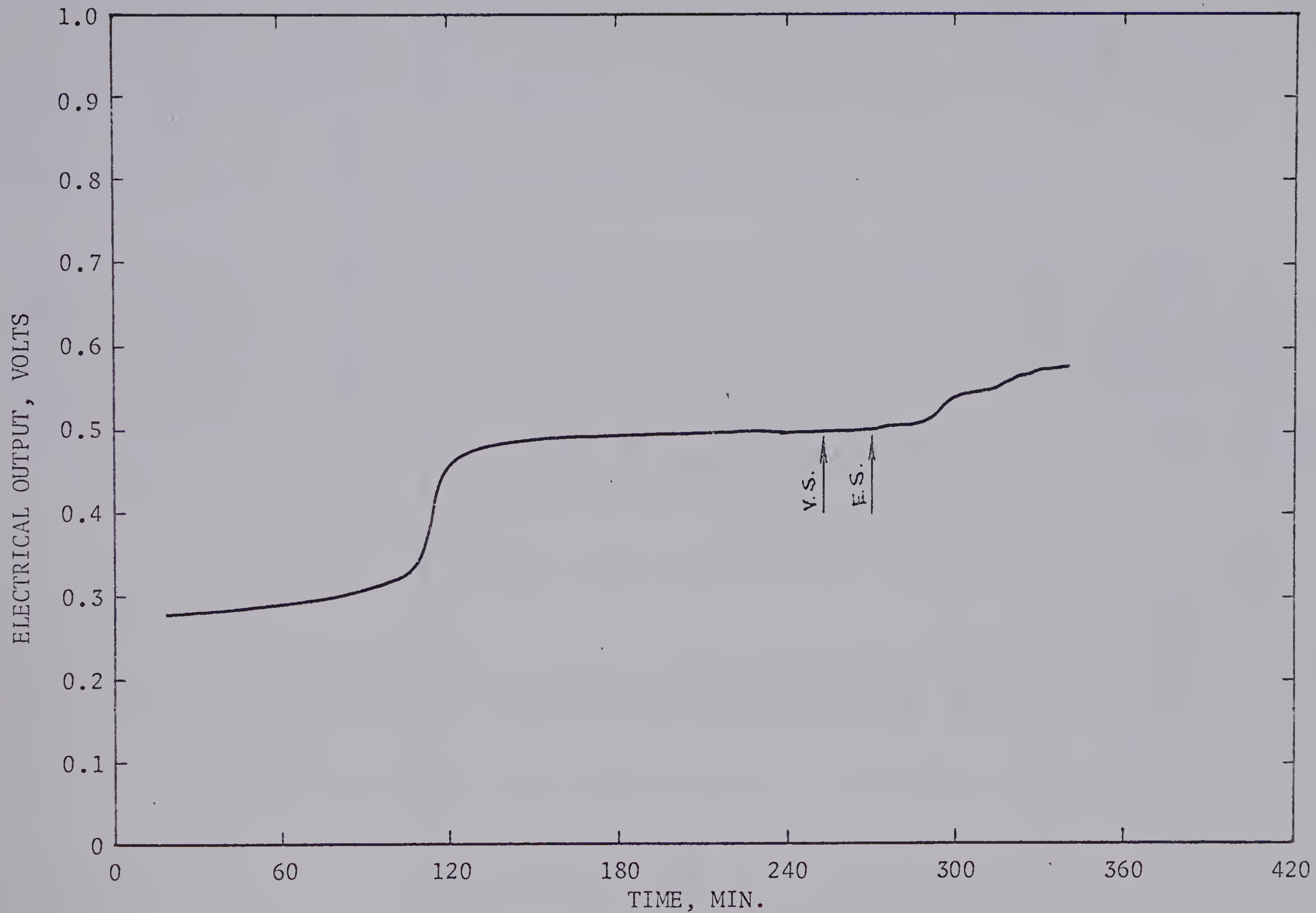

FIGURE V.8 TYPICAL RECORD OF ELECTRICAL TEST; TEST NO. AO 12, TYPE (1)

TABLE V-2

"AC" TYPE ELECTRODE

Test No.	Vicat test results (min.) x_i	R_x	Electrical test results (min.) x'_j	$R_{x'}$
AC 1	295	(19)	270	(11)
2	258	(7)	286	(17)
3	272	(13)	166	(2)
4	281	(16)	328	(22)
5	269	(10)	214	(4)
6	273	(14)	190	(3)
7	267	(8)	316	(20)
8	276	(15)	271	(12)
9	290	(18)	322	(21)
10	245	(5)	68	(1)
11	255	(6)	267	(9)

Results of the Wilcoxon W Test:

T = 131

W = 56

E(W) = 60.50

Var(W) = 23.91

$$\left| \frac{W - E(W)}{\sqrt{Var(W)}} \right| = \underline{0.295 < 1.96}$$

TABLE V-3

"AO" TYPE ELECTRODE

Test No.	Vicat test results (min.)		Electrical test results (min.)	
	x_i	R_x	x'_j	$R_{x'}$
AO 1	285	(25)	278	(21)
2	290	(30)	280	(23)
3	288	(28)	283	(24)
4	277	(19)	336	(42)
5	286	(27)	292	(32)
6	277	(20)	308	(39)
7	266	(8)	265	(6)
8	259	(3)	271	(12)
9	279	(22)	290	(31)
10	276	(16)	307	(38)
11	285	(26)	273	(14)
12	252	(2)	270	(9)
13	261	(5)	276	(17)
14	270	(10)	305	(36)
15	270	(11)	293	(33)
16	260	(4)	289	(29)
17	265	(7)	276	(18)
18	271	(13)	294	(34)

TABLE V-3 -- Continued

Test No.	Vicat test results (min.)		Electrical test results (min.)	
	x_i	R_x	x'_j	$R_{x'}$
AO 19	303	(35)	318	(41)
20	275	(15)	305	(37)
21	250	(1)	312	(40)

Results of the Wilcoxon W Test

$T = 327$

$W = 345$

$E(W) = 220.5$

$Var(W) = 1580.25$

$$\left| \frac{W - E(W)}{\sqrt{Var(W)}} \right| = \underline{3.132 > 1.96}$$

TABLE V-4

"BC" TYPE ELECTRODE

Test No.	Vicat test results (min.)		Electrical test results (min.)	
	x_i	R_x	x'_j	$R_{x'}$
BC 1	272	(6)	277	(10)
2	278	(11)	311	(21)
3	267	(3)	305	(20)
4	283	(14)	256	(2)
5	267	(4)	276	(8)
6	270	(5)	288	(18)
7	243	(1)	286	(16)
8	288	(19)	283	(15)
9	272	(7)	279	(12)
10	276	(9)	348	(22)
11	279	(13)	286	(17)

Results of the Wilcoxon W Test:

$T = 92$

$W = 95$

$E(W) = 60.50$

$Var(W) = 231.91$

$$\left| \frac{W - E(W)}{\sqrt{Var(W)}} \right| = \underline{2.27 > 1.96}$$

TABLE V-5

"BO" TYPE ELECTRODE

Test No.	Vicat test results (min.) x_i	R_x	Electrical test results (min.) x'_j	$R_{x'}$
BO 1	246	(6)	268	(17)
2	235	(3)	198	(2)
3	253	(9)	241	(5)
4	240	(4)	262	(14)
5	258	(11)	256	(10)
6	261	(13)	271	(18)
7	273	(20)	246	(7)
8	258	(12)	279	(22)
9	272	(19)	178	(1)
10	277	(21)	265	(16)
11	264	(15)	250	(8)

Results of the Wilcoxon W Test:

T = 133

W = 54

E(W) = 60.50

Var(W) = 231.91

$$\left| \frac{W - E(W)}{\sqrt{Var(W)}} \right| = \underline{0.426 < 1.96}$$

TABLE V-6

"C" TYPE ELECTRODE

Test No.	Vicat test results (min.) y_{i1}	Electrical test results (min.) y_{i2}	$x_i = y_{i2} - y_{i1}$
C 1	285	288	+ 3
2	250	432	+182
3	264	288	+ 24
4	267	270	+ 3
5	280	306	+ 26

Results of the test of paired comparisons:

$s = 63.12$

$n = 5$

$\overline{x}_i = 47.6$

$$\left| \frac{\overline{x}_i \sqrt{n}}{s} \right| = \underline{1.673 < t_{4;0.025} = 2.776}$$

According to the test of paired comparisons the Vicat and the electrical tests results do not differ significantly.

TABLE V-7

"AO" TYPE ELECTRODE, WATER/CEMENT RATIO 0.357

Test No.	Vicat test results (min.) y_{i1}	Electrical test results (min.) y_{i2}	$x_i = y_{i2} - y_{i1}$
101	305	344	+ 41
102	365	384	+ 19
103	370	330	- 40
104	360	345	- 15
105	360	342	- 18

$s = 32.238$

$n = 5$

$\overline{x}_i = -2.6$

$$\left| \frac{\overline{x}_i \sqrt{n}}{s} \right| = \underline{0.180 < t_{4;0.025} = 2.776}$$

TABLE V-8

"AO" TYPE ELECTRODE, CEMENT MORTAR

Test No.	Penetration resistance test results (min.)		Electrical test results (min.)	
	x_i	R_x	x'_j	$R_{x'}$
M 1				
2				
3	305	(11)	268	(5)
4	345	(21)	279	(6)
5	305	(12)	245	(1)
6	320	(16)		
7	350	(22)	291	(7)
8	330	(19)		
9	305	(13)	261	(9)
10	295	(8)	299	(9)
11	315	(15)		
12	300	(10)	255	(2)
13	320	(17)	267	(4)
14	325	(18)		
15	340	(20)		
16	305	(14)		

Results of the Wilcoxon W Test:

$T = 216$

$W = 217$

$E(W) = 56$

$Var(W) = 214.67$

$$\frac{W - E(W)}{\sqrt{Var(W)}} = \underline{10.99 > 1.96}$$

5.2.4 Discussion of the results

When the results obtained from the Wilcoxon W test are compared against $Z_{\alpha 2}$ which has the value 1.96 at 5 per cent level of significance, the conclusions are as follows:

"AC" type of electrode. Results of the Vicat and the electrical tests do not differ significantly (TABLE V-2).

"AO" type of electrode. Results of the Vicat and the electrical tests do differ significantly and the hypothesis H_0: $F_1(x) \equiv F_2(x)$ is not accepted (TABLE V-3).

"BC" type of electrode. Results of the Vicat and the electrical tests do differ significantly and the hypothesis H_0: $F_1(x) \equiv F_2(x)$ is not accepted (TABLE V-4).

"BO" type of electrode. Results of the Vicat and the electrical tests do not differ significantly (TABLE V-5).

"AO" type of electrode, test on cement mortar. Results of the penetration (standard) and the electrical tests do differ significantly and the hypothesis H_0: $F_1(x) \equiv F_2(x)$ is not accepted (TABLE V-8).

The "C" type electrode tests on cement paste with normal consistency water content and "AO" type electrode tests on cement paste with the water/cement ratio 0.357 were tested by the parametric method of paired comparisons. Conclusions are as follows:

"C" type of electrode. Results of the Vicat and the electrical tests do not differ significantly (TABLE V-6).

"AO" type of electrode, water/cement ratio 0.357. Results of the Vicat and the electrical tests do not differ significantly (TABLE V-7).

Standard deviation was calculated for every set of data to compare the particular types of tests. Calculation was done using the Hewlett-Packard Calculator No. 9100A with program No. 09100-70001, Statistics. Results are listed in TABLE V-9.

TABLE V-9

STANDARD DEVIATIONS AND MEAN VALUE OF THE

ELECTRICAL AND THE VICAT TEST RESULTS

Type of electrode	Electrical test			Vicat test		
	n	$\bar{x}$	s_x	n	$\bar{y}$	s_y
AC	11	245.3	79.3	11	271.0	14.8
AO	21	291.5	18.4	21	273.6	13.3
BC	11	290.5	24.0	11	272.3	11.2
BO	11	246.7	31.4	11	257.9	13.6
AO Mortar	8	270.6	18.1	14	318.6	17.5*
C	5	316.8	65.6	5	269.2	13.9
AO 357	5	349.0	20.5	5	352.0	26.6**

Where n is sample size

$\bar{x}$, y are mean values (min.)

s_x, s_y are standard deviations.

*The penetration resistance test was used.

**Canada Normal Portland Cement, water/cement ratio 0.357

When the standard deviations were taken into consideration and compared, the following electrode types were rejected because of the resulting high standard deviation values.

"AC" type of electrode, s_x = 79.3,

"BO" type of electrode, s_x = 31.3 and

"C" type of electrode, s_x = 65.6.

The remaining tests have standard deviation values very close to those obtained using the Vicat test.

The Wilcoxon W test for two samples shows that the remaining test results ("AO" and "BC" types of electrodes) do differ significantly from the Vicat test results at the 5 per cent level of significance. When the statistical data in TABLE V-9 are compared it is clear that both tests with "AO" and "BC" types of electrodes have very close mean values and similar standard deviations. When these tests are compared on the basis of the null hypothesis, it is indicated that the test results are identical. It is possible to assume, therefore, that both electrodes have indicated the same change of properties of tested cement paste.

The cement paste characteristic indicated by the electrical change occurs at a time close to the initial setting time of cement as indicated by the Vicat test. The fact that the two times do not coincide is of no significance since the properties measured by the two tests are not the same. The Vicat test in effect measures some strength characteristic of the setting paste and the Vicat initial setting time indicates the time that the cement paste has reached some arbitrary value of strength. On the other hand, the electrical test determines the time of some change in the voltage output of the electrical cell. The fact that the two results are close is strictly coincidental, but this fact also offers the possibility of an alternative method of defining setting time and provides a procedure for its determination. It is important also that such an alternative method provide adequate accuracy and reproducibility of results. The electrical test seems to provide such accuracy and reproducibility.

Most of the tests were done on cement pastes mixed to normal consistency. When the water/cement ratio was changed, the electrical test results were also changed, in the same way as were the Vicat test results. Higher water cement ratios delayed the setting time of the cement pastes.

When cement mortars were tested, the electrical output (voltage) indicated the characteristic of setting time in only 8 tests out of 16 as listed in TABLE IV-8. The electrically determined time of test, where it could be determined, was on the average less than that indicated by the penetration resistance test. If an inert aggregate is assumed the hydration reactions of cement paste in mortar would not be expected to be affected by the aggregate presence appreciably. One possible change of conditions was that aggregate heat accumulation lowered the temperature of the mixture.

With respect to the penetration test results, the setting time values varied considerably depending on the size of penetration needle that was used. The difference obtained by using two or three different sizes of needles at the same time was as high as 35 per cent. To overcome this difficulty, a needle size of one tenth of a square inch was consistently used to establish the time of set by the penetration resistance method.

CHAPTER VI

CONCLUSIONS AND RECOMMENDATIONS

6.1 Conclusions

Two different methods were tested during this investigation to study the behaviour of Portland cement paste during the plastic state and into the hardened state; (1) the continuous workability test for testing the pre-setting properties of a plastic cement paste and (2) the continuous electrical test for testing setting properties of fresh cement paste and cement mortar. Conclusions and limitations are as follows:

The workability test

(a) gives different characteristic workability curves for different types and sources of cements,

(b) indicates a varying sensitivity to water content for different types and sources of cements.

Limitations of the workability test:

(a) workability is tested on continuously moving sample of cement paste thus breaking its structure that develops during hydration process,

(b) narrow range of water/cement ratios that the testing equipment can handle.

The electrical test

(a) shows that a "setting time" can be determined on the basis

of one of the three rules listed in section 5.2.1,

(b) indicates that its result as listed here are sufficiently accurate and closely related to the "initial setting time" as determined by the Vicat test to warrant its consideration as an alternative to the Vicat time of set test,

(c) gives reproducibility that is of the same order as that of the Vicat test.

Limitations of the electrical test research program:

(a) results of electrical test were compared with the Vicat test results,

(b) limited number of sources of cement that could be properly tested due to limited time.

6.2 Recommendations

The workability test:

(a) further studies are necessary to obtain information to define the term of workability more precisely,

(b) it is necessary on the basis of studies in (a) to relate laboratory investigations to the concept of "workability" as used by the workmen in the field.

The electrical test:

(a) further studies are necessary to remove the limitations and to expand the possibilities of the electrical test,

(b) it is necessary to compare the electrical test results with existing methods that enable a continuous observation of the setting characteristic of cement paste such as X-ray

continuous diffraction (Seligmann and Greening, 1964) in order to fully explain the results obtained.

(c) It is necessary to study the theoretical basis to explain the nature and origin of the electrical output changes.

(d) Since the electrical test on cement mortars did not achieve the same results on cement paste, further studies should be carried out. Modifications of the equipment and the use of different types of electrodes should be considered.

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ASTM C 305-65, Standard Specifications for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency.

ASTM C 403-63T, Tentative Method of Test for Time of Setting of Concrete Mixtures by Penetration Resistance.

ASTM D 1558-63, Standard Method of Test for Moisture - Penetration Resistance Relations of Fine-Grained Soils.

Appendix A

THE HALLIBURTON THICKENING

TIME TESTER

THE HALLIBURTON THICKENING TIME TESTER

Model 800.5

I Test

Test of cementing slurries prepared from hydraulic cement intended for subsurface oil well cementing uses.

II Purpose

1. To provide a practical field or laboratory means of evaluating a hydraulic cement for a particular type of oil well cementing operation.

2. To provide a practical field or laboratory means for a direct comparison of two different cements under identical conditions of temperature and agitation.

3. To provide a practical field or laboratory means for a study of the setting characteristics and viscosity of various cements and additives as related to their use in subsurface oil well cementing.

III Apparatus

The apparatus consists of a stainless steel case having two compartments. The left compartment is the consistometer proper containing

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*These instructions are offered as a guide and explanation for the operation of the Halliburton Thickening Time Tester, Model 800.5. They do not apply to the operation of the Halliburton Cement Consistometer, Model 800.0, which was manufactured and sold by the Halliburton Oil Well Cementing Company, previously to 1950.

the constant temperature bath, slurry containers and container driving mechanism. The right compartment houses the motor and drive pulley.

The two-slurry containers are suitably mounted for rotation inside the bath which may be regulated to any desired temperature between 80°F and 200°F. The slurry containers and paddles are of stainless steel. The tops of the cylinders are fabricated of phenolic plastic with the spindle and torque drum of stainless steel. A single row, double shield ball bearing supports the spindle and drum.

The slurry container mounts and the driving mechanism are attached to the top of the case. The container driving worm is made of carbon steel, the worm gear is of bronze, and the drive shaft is of stainless steel.

The containers are rotated by engaging the pins extending from the top with the grooves of the support. The support has a gear around it which is driven by the worm. The motor should be stopped momentarily while disengaging the container from the support.

The thermoregulator knob for adjusting the temperature is on the front of the right compartment. This knob is connected by means of a flexible shaft to the thermoregulator protruding into the bath.

In the front of the consistometer compartment is the fluid level gauge. When the machine is operating, the fluid level should be so that the gauge is filled approximately one-half full.

The torque indicating mechanism is both simple and reliable. The inherent inaccuracies of springs as used in graduated pull indicating devices are avoided by the adoption of a simple pendulum construction. As the pendulum is pulled from the vertical a moment is created.

The value of this moment varies from zero to a value equal to the weight of the stainless weight plus the weight of the lever arm multiplied by the perpendicular distance out to the center of gravity of the stainless weight and the lever arm considered as a unit. The quadrant scale in back of the pointer is graduated in ten equal increments of torque, although the angular distance between successive graduations varies. The torque indicating device is connected to the drum on top of the slurry container by a string.

The small motor which drives the machine is of the split phase type, and is controlled by a switch on the base board. Power is transmitted by a small V belt.

Heat is supplied by means of two cal rod heating units of 600 watts each. The units are connected to a 3-way switch mounted on the front of the instrument. By turning this switch to LOW, MEDIUM, or HIGH the units are connected in parallel, only one heater, or in series, respectively. This gives a selection of three rates of heating, namely, 300 watts, 600 watts or 1200 watts. The thermoregulator is connected into the heating circuit at all heating positions, but the life of the thermoregulator will be materially extended if the switch is turned to LOW or MEDIUM as required, as soon as the desired temperature has been attained. The desired temperature is maintained by adjusting the thermoregulator knob so that the light above it will turn on and off plus and minus one degree of the proper temperature.

The ball bearings, four in all, two in the tops of the cylinders and two in the torque indicating mechanism, are of the double shield type to eliminate all possible foreign matter. Dirt in the bearings may

be detected readily by the "rough" feel when rotated with the hand.

There are two lubricators on top of the case which lubricate the drive shaft bearings. Care should be taken to be sure that these points are well lubricated. Also, oil should periodically be put around the slurry container supports which lubricates the worm and gear. The motor may be oiled by removing the perforated disc at the back of the instrument.

*IV Preparation of Sample

The cement sample, assumed to be representative, should be passed through a #20 standard sieve to eliminate any unground clinker that may be present.

V Procedure

The paddle and bearings should be tested for excessive friction by running the cylinder assembled, but without cement inside, for a few minutes. If the paddle is bent to such an extent that it rubs on the side, or if the bearing is in need of cleaning, an appreciable movement of the torque will be noted. These defects should be corrected before any tests are started.

Each cylinder of the consistometer has been designed to contain 500 cc with some space left at the top and the height to which 500 cc rises has been marked inside the cylinder. The paddle is replaced, and the lid put on, being careful that the slotted shaft engages the pin in

*IV Preparation of Sample - (See Section II "Preparation of Slurry" of API RP10B).

the top. The cylinder is then placed in the consistometer, and the torque indicator string passed around the knob and looped over the small screw. The string must lie along the outside of both the torque indicator ring and the knob, in order to preserve a constant radius of pull. With the consistometer running, a certain reading will be indicated on the torque indicator scale.

Complete procedures for preparing the sample, selecting a water-cement ratio, mixing the slurry, performing the tests and recording the results are described in API Bulletin RP 10B "Recommended Practices for Testing Oil Well Cements." Where practical, these standard tests should be performed as outlined for proper correlation of test data from various laboratories.

VI Calculation of Results

The results of tests are read directly from the indicator and no calculations are necessary so long as the machine is in calibration.

However, the consistency readings may be plotted on common graph paper with the consistency as the ordinate and time as the abscissa.

VII Interpretation of Results

The general shape of the consistency-time curve plotted as described above presents an excellent picture of a particular cement as far as its setting characteristics are concerned. A visual comparison of the curves of several cements will usually disclose characteristic differences of more or less importance when considered in relation to particular cementing application.

There are certain features common to all consistency-time curves.

As the cement is introduced, it generally has a fairly low consistency. As stirring is commenced, this value usually drops a little more. It then commences to increase at a very gradual rate. However, as time goes on, the rate of increase of consistency is accelerated to such an extent that the latter part of the curve is quite steep. This acceleration varies with different cements and with different temperatures of testing.

From the standpoint of the majority of oil well cementing requirements, a proper cement slurry should reveal the following characteristics on a consistency curve produced with a Halliburton Consistometer at temperatures corresponding to well conditions.

1. Initial consistency between 10 and 30 poises.
2. Consistency to remain below 40 poises for 3 hours from time of introduction into consistometer.
3. Sharp break toward higher consistency after three hours.
4. Tangent to curve should be practically vertical when the curve reaches consistency of 10. This feature is indicative of the rapidity with which the cement will develop strength after initial set.

NOTE: It is understood that units of measurement of viscosity should properly apply only to true viscous fluids, and that cement slurry is not a true viscous fluid; however, the effective resistance to agitation offered by a cement slurry can be conveniently measured by a Halliburton Consistometer, which also measures the viscosity of a viscous fluid; hence, the advantage of expressing the consistency of a cement or a mud slurry in terms of a universally known unit such as a poise.

VIII Calibration Procedure - (See Sec. VII API RP 10B paragraph 50)

This procedure is intended for calibration of the equipment so that readings on the index of torque will indicate poises -- a unit of viscosity. The index of torque is divided into ten divisions with each division representing 10 poises. The oil used in the calibration process is called Paratone, having a viscosity which is known over a range of 5 to 100 poises. The Paratone calibration oil may be obtained from the Halliburton Oil Well Cementing Co., at Duncan, Okla., under part number 70.30962.

Before beginning calibration, the apparatus should be leveled and the indicating pointer set to read zero when the pendulum hangs free with the string lying on top of the consistometer case. Without any fluid in the slurry containers, the string should be attached and the motor started to determine whether the indicating mechanism will remain at zero. This indicates lack of measurable friction in the bearings and proper alignment of the bearings. If the indicator does not remain steady at zero, realignment or replacement of the bearings should correct this improper function.

Following the above operations, each slurry container should be completely dry before pouring the Paratone into them. The paddle should then be placed in the container and the covers properly adjusted. The bath on the apparatus should be filled with water so that when the slurry containers are placed in the bath the water level will not rise past the center of the level gauge.

With the slurry containers in place, the string of the indicating mechanism attached to the cover and the motor started, the heater should

be turned on and the thermoregulator adjusted to maintain the proper temperature for a Paratone viscosity of 20 poises. The correct temperature is obtained from the curve which accompanies the Paratone oil. After this temperature has been reached, it should be maintained for a period of one hour to insure complete equalization of temperature within the slurry container. After one hour, if the viscosity reading on the index scale does not correspond with the 20 poise mark (index reading 2), the bob on the lower end of the pendulum should be adjusted either up or down so that the reading will be correct. Under no condition should the indicator pointer be moved after previously having been set at zero.

Following establishment of the bob position for a 20 poise viscosity, checks should be made at temperatures corresponding to approximately 10, 50, and 80 poises to determine what deviations might be expected throughout the range of the index scale. For these measurements it should only be necessary to maintain the constant temperature for 30 minutes to obtain equalization throughout the Paratone.

After readings from the index quadrant have been recorded at the various temperatures, the Paratone should be discarded and new material used for calibrating subsequent machines. This procedure is that specified by API in bulletin RP 10B, "Recommended Practices for Testing Oil Well Cements."

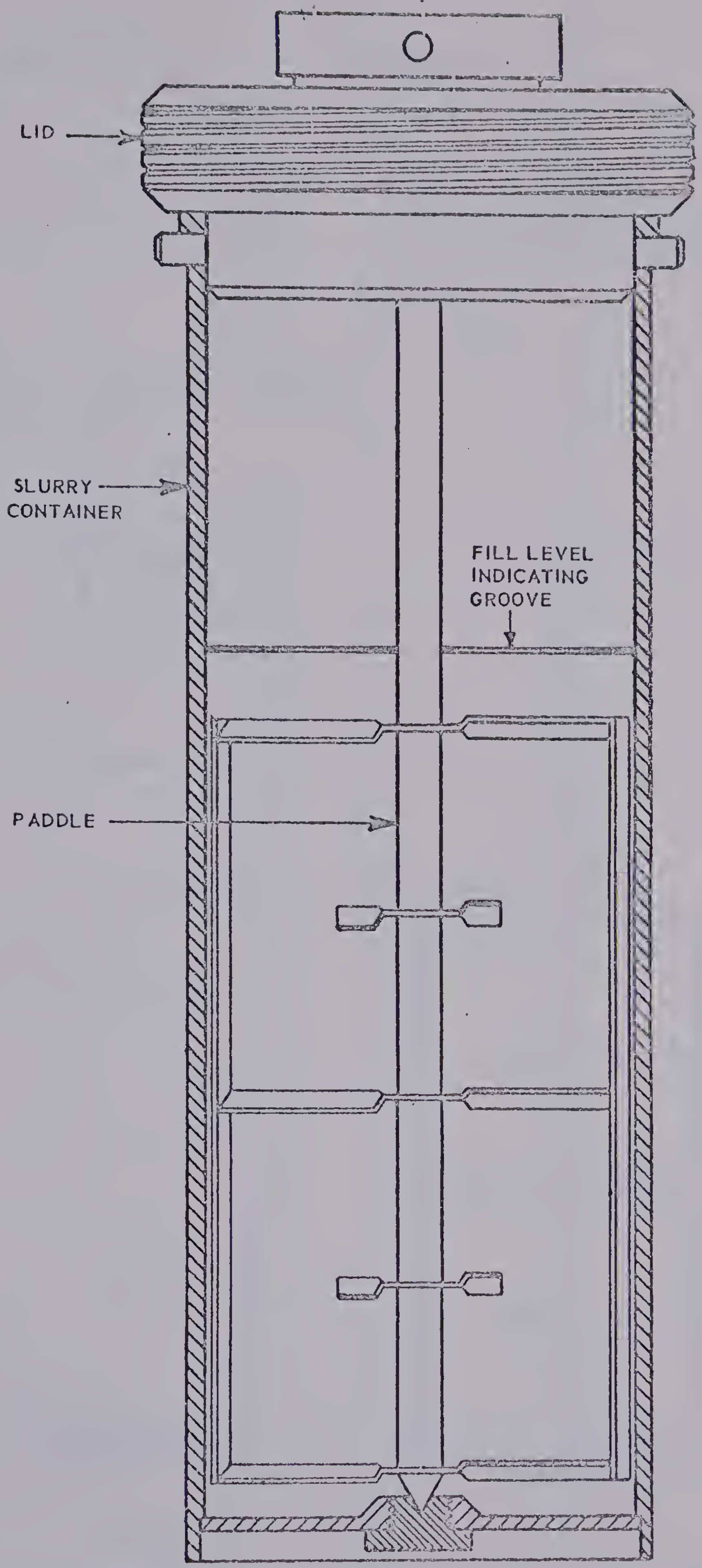
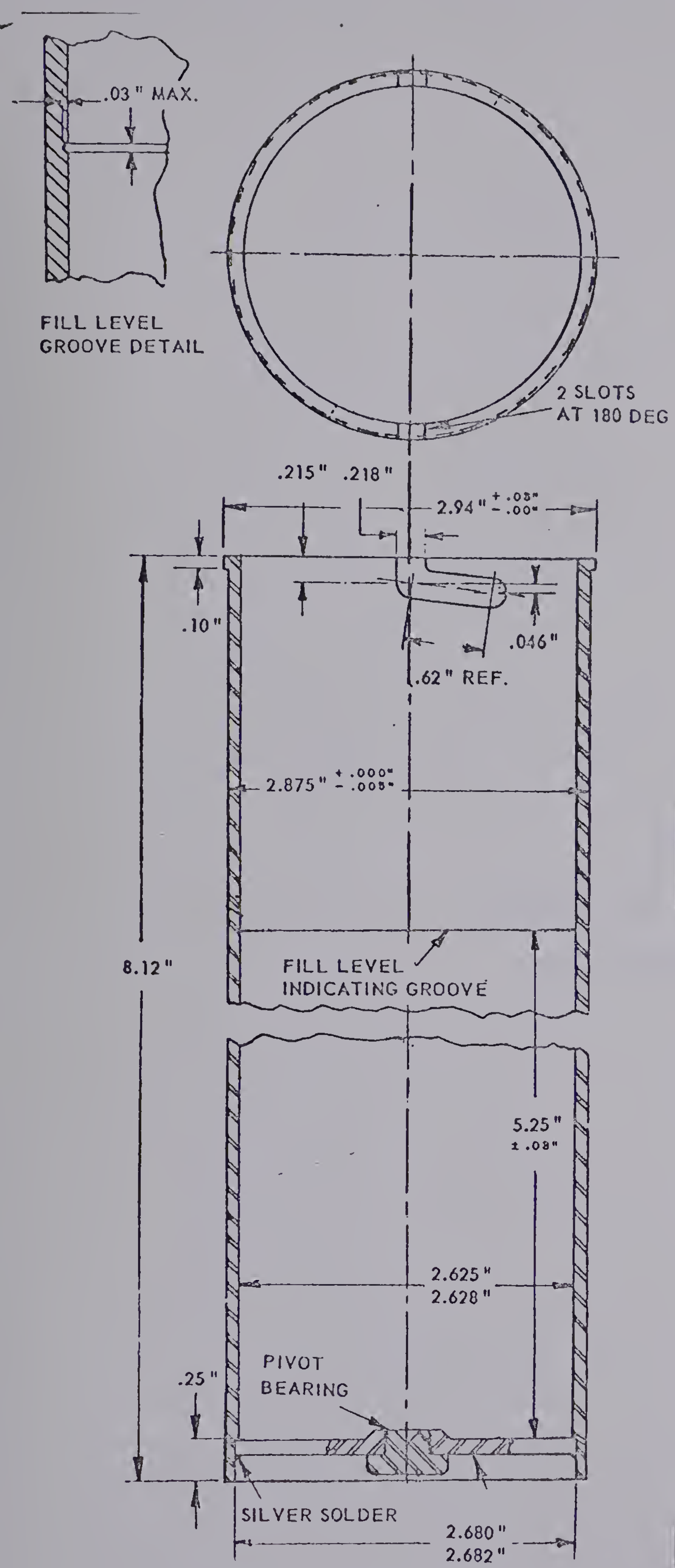
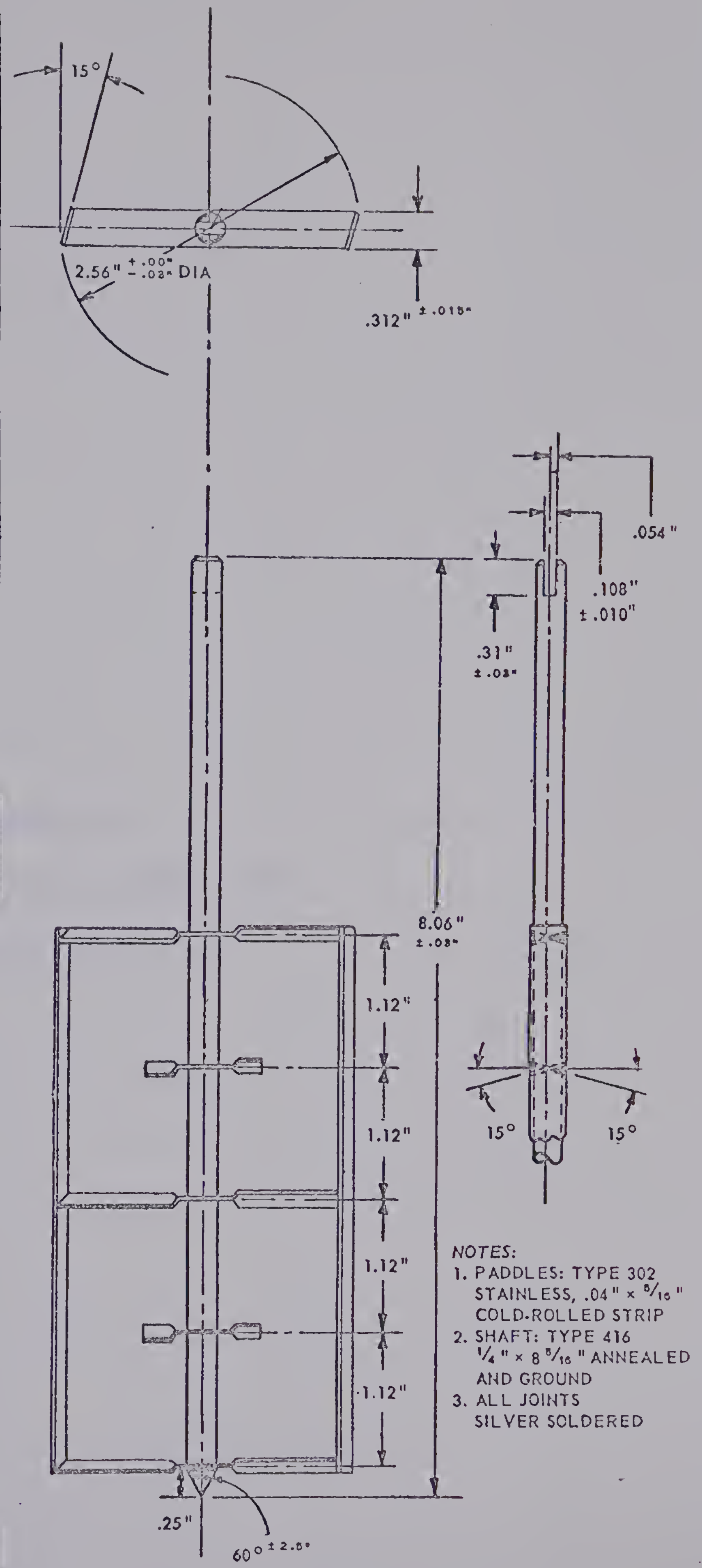


FIGURE A.1 HALLIBURTON CONSISTOMETER ASSEMBLY



SLURRY CONTAINER
HALLIBURTON CONSISTOMETER

FIGURE A.3



PADDLE
HALLIBURTON CONSISTOMETER

FIGURE A.2

Appendix B

DESCRIPTION OF ELECTRODES USED FOR THE ELECTRICAL TEST

DESCRIPTION OF ELECTRODES USED
FOR THE ELECTRICAL TEST

Five types of electrodes were used during the electrical testing procedure. The purpose was to develop and select the best performing electrode for further testing. Three basic types of electrodes, according to their shape, were involved in the investigation; type A, type B and type C. The types A and B were further modified to a type with an "open face" - "AO" and "BO" type electrodes - and to a type with a "closed face" - "AC" and "BC" type of electrodes. "Closed face" electrodes were completely insulated except the "active part" that was completely immersed into the coment paste. The reason for "open" and "closed face" and different shapes of electrodes was to find if the air penetration into a hardened cement paste had any influence on the electrode's performance. Two different types of electrodes, "AO" and "BC" showed best results as listed in Chapter V. This may indicate that the air penetration probably did not influence the function and performance of the electrodes.

The electrodes are shown in FIGURES B1, B2, B3, B4 and B5.

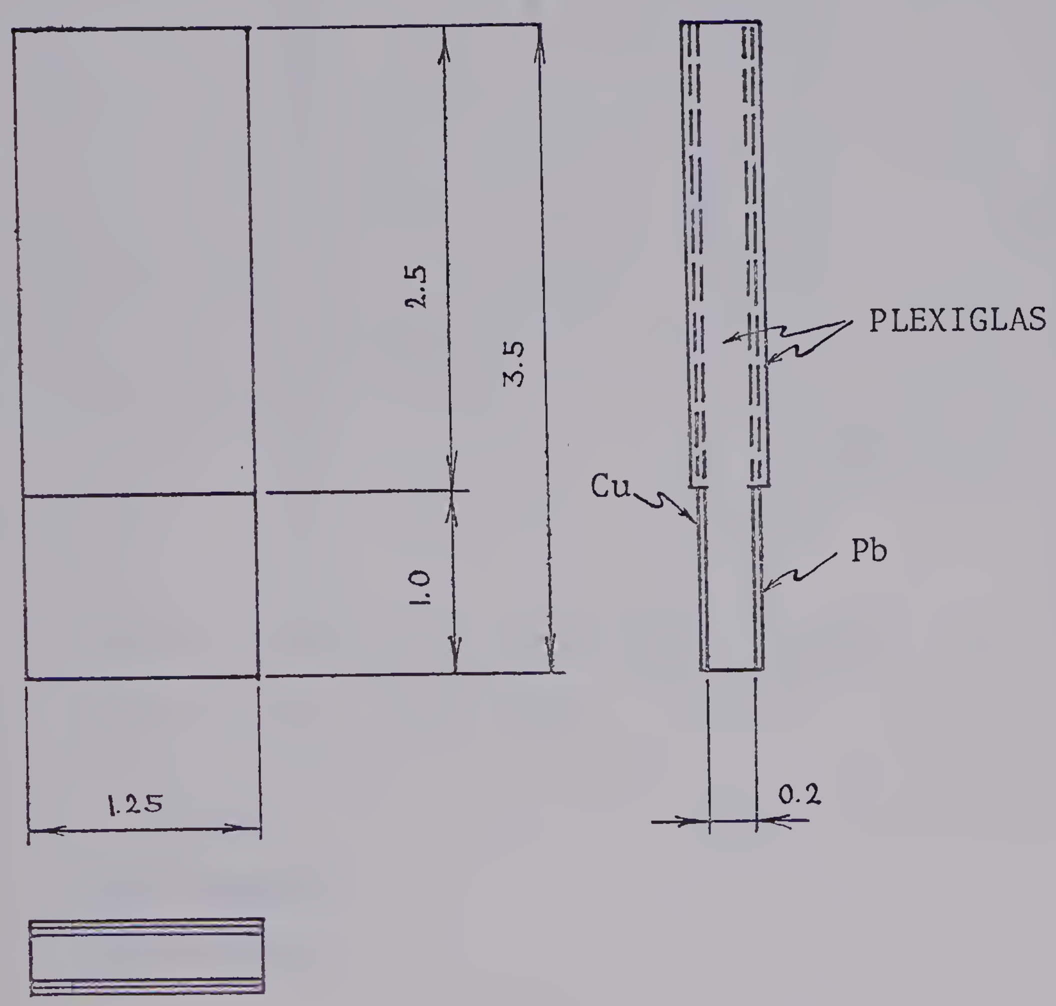


FIGURE B.1 "AC" TYPE ELECTRODE

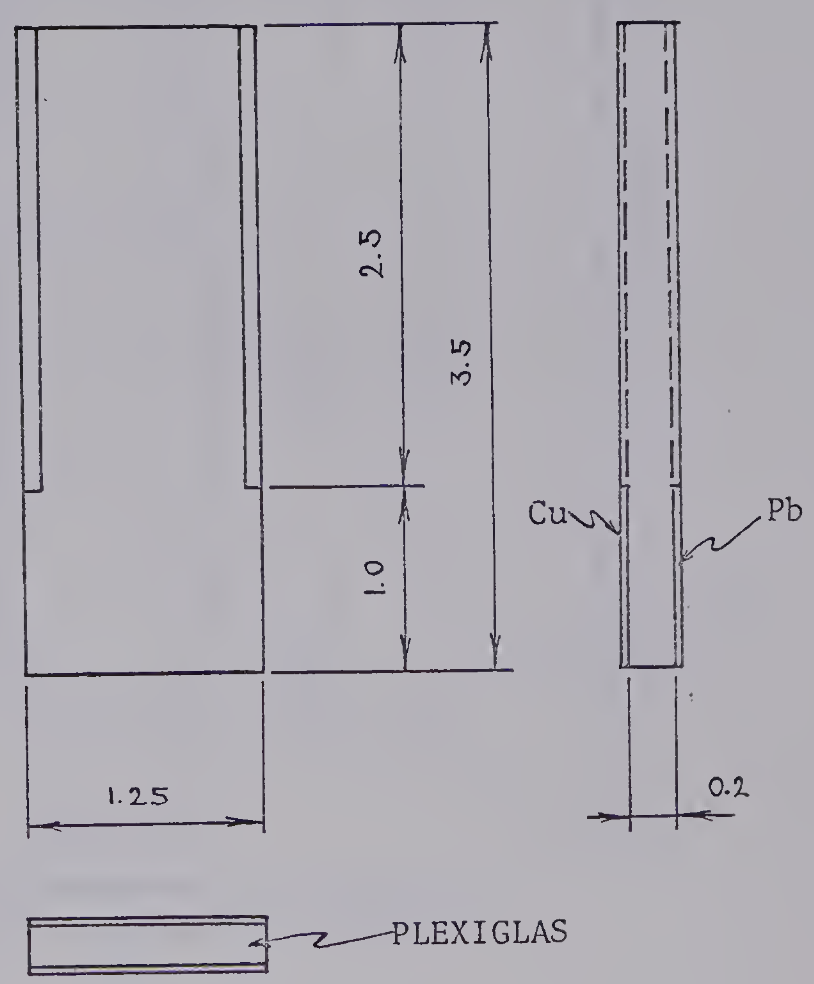


FIGURE B.2 "BC" TYPE ELECTRODE

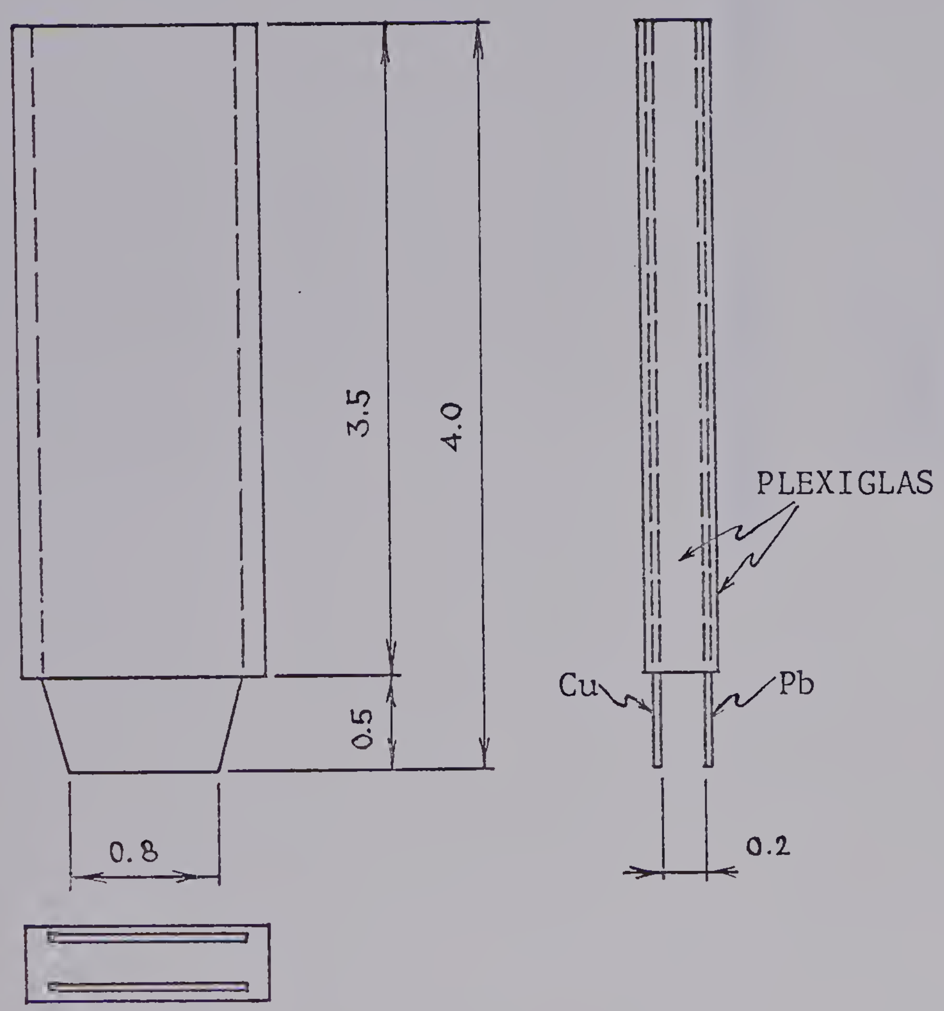


FIGURE B.3 "BC" TYPE ELECTRODE

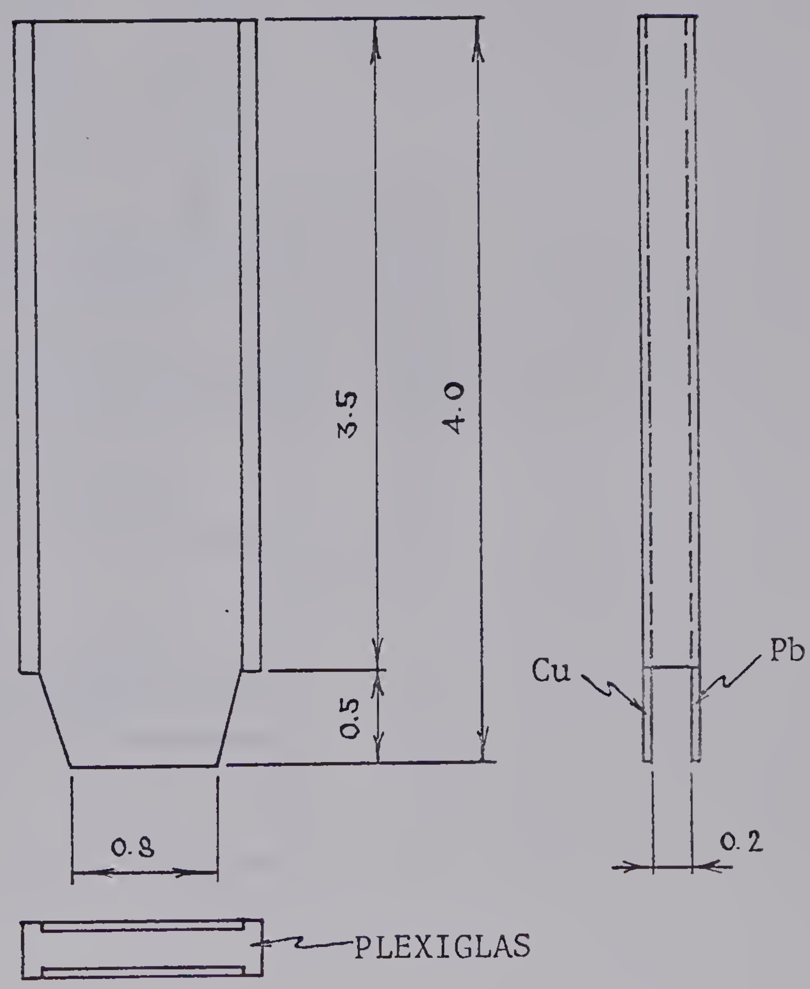


FIGURE B.4 "BO" TYPE ELECTRODE

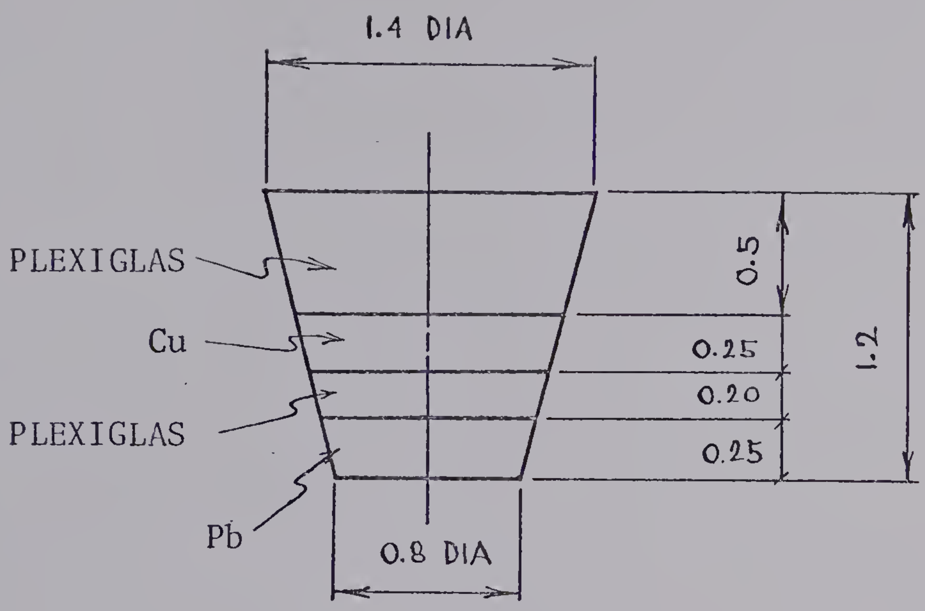


FIGURE B.5 "C" TYPE ELECTRODE